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# Micromorphological and mineralogical features of saline playa surface sediments from two large Trans-Uralian lakes

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# Abstract

The proposed paper is devoted to the features of mineralogical composition and microstructure of saline playa surface sediments from two large drainless lakes in the south west of Western Siberia. The material composing the surface sediments of both playas is an unlithified mixture of clay and sand, with a significant admixture of organic matter. Coarse material is represented mainly by quartz, with an insignificant admixture of feldspar grains and micaceous fragments. In general, terrigenous component is characterized by a comparatively low degree of sorting. Clay material is scarce and composed, presumably of chlorite-hydromica material with a significant admixture of undecomposed organic matter, and traces of ferruginization. Carbonates and evaporates are the most common authigenic minerals. In both cases carbonates occur as microconcretions that correspond to the zone enriched with cysts, plant detritus and other degrading organic matter. Evaporates occur both as the efflorescence on the surfaces of the crusts. The study results have shown that surface crusts contain zones enriched with Artemia salina cysts, which are a significant component of sediments. Degrading crusts promote secondary mineral formation, especially formation of carbonates. Surface crusts of two studied playa environments differ in proportion of terrigenous material, clay minerals, as well as the composition of evaporates and carbonates.

Keywords: Artemia salina, lakes, Solonchaks, Western Siberia.

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# Introduction

Small and large lakes, that significantly vary in terms of water chemistry and morphometric parameters are important components of the landscape mosaic in the arid and semiarid regions of Western Siberia and Northern Kazakhstan (Yermolayev and Wizer, 2010). A significant proportion of this water bodies is undrained and rather often saline (Ovdina et al., 2020), which results in the formation of specific aquatic and subaquatic ecosystems adjacent to such environments (Zarubina and Durnikin, 2005; Samylina et al., 2014). Another important feature of the lakes within the territory under consideration is related to the fact that they are very dynamic systems, subjected to significant seasonal, annual and decade-long fluctuations of water level (Meyer et al., 2008; Rudaya et al., 2012), that can result in their full drying off. Global and regional natural and human-induced transformation of the climatic conditions and landscapes can intensify the degradation of these water bodies and even make this process irreversible.

Large saline lakes within the steppe and forest-steppe zone of Western Siberia and Northern Kazakhstan form solonchakous playas around them. During the gradual drying of these water bodies bottom sedimets are exposing to the land surface and are subjected to the processes of the initial soil formation (Kazantsev et



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al., 2005). On the one hand, these surfaces can play a potentially important role for the carbon sequestration (Yakutin et al., 2016a,b); on the other hand, a number of studies have shown that dry environments can also act as sources of  $CO_2$  emissions (Beltrán-Hernández et al., 2007; Ikkonen et al., 2018). It is also worth mentioning, that degradation of saline lakes leads to the formation of immense areas covered with salt crusts, that can act as potential sources of salt-rich dust, and, consequently, lead to further salinization of the adjacent agricultural landscapes (Mees and Singer, 2006).

The above arguments caused a consistent interest to the studies devoted to the biogeochemistry, minerology, properties and microstructure of near-shore *Solonchaks*, and salt crusts covering dry areas of the lake beds (Mees and Singer, 2006). Various studies have been performed for such lake-playa environments in different arid and semi-arid regions of the world (Hammer, 1986; Vizcayno et al., 1995; Castañeda et al., 2005, 2015; Joeckel and Clement, 2005; Castañedaa and Herrero, 2008; Mees et al., 2011; Gutiérrez et al., 2013; Zhang et al., 2013; Acree et al., 2019; Smolentseva and Gavrilov, 2020; Jafarpoor et al., 2021). Within the south of Western Siberia and Northern Kazakhstan near-shore *Solonchaks* and salt crusts were extensively studied for the territories of the Kulunda and central Baraba regions (Lebedeva et al., 2008; Yakutin et al., 2016a). At the same time, for the Trans-Uralian, the south-western part of the Western Siberia, similar studies are much less common; saline landscapes and soils within the territory under consideration are rather poorly studied.

The largest areas occupied by salt playas within the territory of the Trans-Urals are confined to a number of lakes located in the Kurgan region on the border with Kazakhstan (lakes Medvezhye, Sazykul, Gorkoe, Siverga, Elanch, Kaltyk, etc.) (Shulpina, 2004). Investigation of the soils and surface crusts of large saline lakes within study area is important for better understanding the functioning of these systems that experience intense anthropogenic impact associated with a significant recreational load. Resorts, balneological sanatoriums, recreation sites, as well as areas with developed brine shrimp gathering within the region are, as a rule, confined to large saline water bodies.

This work presents the results of a study of the features of the microstructure and mineral composition of the surface sediments of large playas within lakes Medvezhye and Kaltyk.

# **Material and Methods**

#### Study area and sampling

The studied lakes are situated in the south-western part of the West Siberia Plain within the Tobol-Irtysh interfluve. Lakes Kaltyk and Medvezhye are located in Kurgan Oblast, in the Lebyazhyevsky and Petukhovsky districts, respectively (Figure 1). The territory is characterized by a predominance of low slightly-dissected relief with absolute heights from 120 to 140 m above sea level. Undrained lakes and forest-steppe bogs occupy local depressions. The covering deposits are represented by Late Quaternary carbonaceous loess-like loams, which are underlain by Neogene and Oligocene continental sediments (Novoselov et al., 2019).

The climate of the territory is continental, typical for the forest-steppe subzone of the Southern Urals, with low precipitation, hot summer and cold winter seasons. The annual temperature is 2.7 °C, the annual precipitation is less than 400 mm (Shulpina, 2004). The vegetation of the territory under consideration is strongly transformed by agricultural activities. The typical landscape pattern is characterized by the alteration of ploughed fields with *Luvic Chernozems*, beach forests with *Luvic Phaeozems* and *Mollic Planosols*, as well as *Solonetz* and *Solonchaks* soils under halomorphic vegetation within the areas adjacent to saline lakes.

Field studies were carried out in July 2018 within the areas of playa landscapes in the northern part of Lake Medvezhye and the southern part of Lake Kaltyk.

Lake Medvezhye is one of the largest reservoirs in the Kurgan Oblast. The total length of the coastal zone of the lake is about 60 km, the average depth is 0.5-1.0 m. Sulfide salt-saturated sulfide-silt therapeutic sapropel forms a 0.7 m thick layer in the bottom of the water body (Kurochkin et al., 2014). The brine of the lake has a chloride-sodium composition and mineralization varying in a wide range of 112-350 g L<sup>-1</sup>. Lake Medvezhye is a large habitat for the *Artemia salina*, which is valuable biological resource (Kurochkin et al., 2014; Litvinenko et al., 2015). The Kaltyk has a similar morphology of the lake bed and is also surrounded by vast saline playas. At the same time, this reservoir is much less studied and is practically not mentioned in the works devoted to the water bodies of the region.

Samples for mineralogical and microscopic studies were collected from different facies of the studied environments.



Figure 1. The location of the studied lakes in the southwest of Western Siberia (satellite images from Google Maps). Red triangles denote sampling sites

#### Mineralogical and microscopic studies

The primary diagnostics of collected samples was carried out using a Leica EZ4 D stereomicroscope (Leica Microsystems, Wetzlar, Germany) with an integrated digital camera. The studies of most representative samples were performed in thin sections using an Eclipse LV100POL polarization microscope (Nikon, Tokyo, Japan). The SEM-EDS analysis was performed using a TM3000 scanning electron microscope (Hitachi, Tokyo, Japan) with a Quantax 70 EDS attachment (Bruker, Billerica, MA, USA) at X100–5000 magnification and a JSM-6390LV scanning electron microscope (Jeol, Tokyo, Japan) with an INCA Energy 450 X-Max80 EDS attachment (Oxford Instruments, Abingdon, UK). The SEM observation were made under high vacuum (HV-mode), mainly in the elemental composition mode (BSE, registration of back scattered electrons). While performing the EDS analysis, the voltage was 15 and 20 kV for the first and second devices, respectively.

The XRD analysis of representative samples from both lakes was performed using Shimadzu XRD-6000C X-ray diffractometer (CuKα radiation, Ni filtr, 30kV, 20 mA).

## **Results and Discussion**

The saline playas of Lake Medvezhye area are widespread within the northern and northeastern shores of the reservoir (Figure 1), where they appear as an isolated shallow liman-like creek filled with water during the high-water period (Figure 2a). The territory of the playa along the entire channel is covered with a fissured dense solid crust, up to 3-4 cm thick, covered with numerous mineral (evaporitic) efflorescences on its surfaces (Figure 2b). The hard crust of the saline playa overlaps the loose moist finely dispersed dark brown sediment without any pronounced stratification and signs of active processes of sulfate-reduction. Interlayers of light red color formed under a dry Takyric crust are characteristic for the peripheral parts of the playa located in the vicinity of the coastal area of the lake. These interlayers are composed from a mixture of coarse mineral material and *A. salina* cysts, mineralized by various degree (Figure 2c,d). Cysts also occur on the surface of the crust, transported by wind and periodic water currents. The largest number of cysts was characteristic for the inshore sediments, were a layer organic capsules cover about 40-50% of the coastal surface.



Figure 2. The saline playas of Lake Medvezhye: inshore zone covered with creamy mass of *A. salina* cysts (a); thin dark layer with sulfate-reduction processes under a dense Takyric crust with efflorescences of salts on its surfaces in the drained part of saline playa (b); thin sections (PPL) illustrating the main mineral components and microstructure of the surface sediments. It is clearly visible that they represent a mixture of terrigenous mineral grains, authigenic pelitomorphic carbonate aggregates, mineralized and slightly mineralized organic remains, including abundant of *A. salina* cysts (c and d)

Lake Kaltyk has simpler configuration of the shoreline, as well as the water body and its basin have a round shape. Large saline playa landscapes appear in the western and eastern peripheral parts of the lake (Figure 1). In the south-eastern part of the water body saline playas correspond to the large creek, which most likely correspond to the outlet of the drained stream, cutting into the adjacent arable land (Figure 1b). The main vegetation of playas is represented by *Salicornia europaea* (Figure 3a), a plant in the described range, which is confined to sea coasts with increased soil salinity, periodic flooding with salt water and a lack of oxygen in the soil. The playas of the Kaltyk have a flat, rather wet surface, complicated by large, rounded, sometimes bowl-shaped, spots of dry cracked crusts, about 2-3 cm thick and up to 5-6 m in diameter (Figure 3b,c). Such areas are often covered with efflorescences of evaporitic minerals (Figure 3d,e,f).

Based on the results of the semiquantative estimation of the mineral composition of the surface crusts from Lakes Medvezhye and Kaltyk by X-ray structural analysis it is possible to note the differences characteristic for these two playa environments.



Figure 3. The saline playas of Lake Kaltyk: peripheral part of drained playa occupied by halophilic vegetation (a); dried spots with takyric crusts with efflorescences of evaporitic minerals (b and c); thin section (XPL), illustrating the mineral composition of surface sediments, in the photo there are dense carbonate microaggregates and non-mineralized organic matter (d); thin sections (PPL and XPL) characterizing the main features of microstructure of playa crusts, represented by an alterations of terrigenous material, carbonate microaggregates and slightly mineralized organic matter (e and f)

First of all, one can see that the studies playas differ in terms of the composition of the terrigenous material. The samples from Lake Medvezhye contain a high proportion of quartz and feldspars, that often exceeds 80%, while for the samples, representing Lake Kaltyk this proportion was much lower, up to 26.5%. However, it should be noted that the sediments of Lake Kaltyk also contain a significantly larger amount of allothigenic clay minerals (mainly illite and chlorite).

Carbonates are rather common among authigenic minerals of surface crusts from both saline playa environments. The main carbonate mineral of the sediments of Lake Kaltyk was aragonite (up to 40.9%), partly of authigenic nature, partly, corresponding to external skeletons of benthic organisms. The composition of carbonates of Lake Medvezhye was more diverse. Siderite, which likely indicates the presence of a reducing conditions during the periods of anaerobic functioning of the playas, calcite (up to 2.5%) and Mg-calcite were also abundant in all analyzed samples.

Gypsum was the most common evaporitic mineral in the surface sediments of both playa environments (Lake Medvezhye – up to 3.7%, Lake Kaltyk – up to 9.9%). It is also worth noting that in the salt marsh sediments on Lake Medvezhye, halite is contained only in the form of thin microcrystalline films on the

surface of the soil crust, while in the salt marsh sediments of Lake Kaltyk, halite is contained directly in the sediment layer, accounting for up to 20.8% of the total mass of mineral matter.

Results of microscopic studies showed that the terrigenous component of the deposits of Lake Medvezhye is represented mainly by the fine sand fraction, with the predominant grain size 0.1-0.15 mm. The grading is medium, single grains have slight traces of dissolution. Quartz grains are semi-rounded with absence of cracks on their surfaces. Rare grains of feldspars also show minor traces of dissolution and pelitization. Clay minerals, as a rule, are hydrated and partially replaced by authigenic carbonates. Authigenic minerals are mainly represented by pelitomorphic carbonates (calcite, Mg-calcite, and siderite), which form microcrystalline concretions, rarely forming rounded microconcretions (Figure 4a). Basically, carbonates are formed along pelitized, hydrated grains, as well as organic matter - EPS films, decaying plant debris, and cysts of *A. salina* cysts (Figure 4d).



Figure 4. SEM photos of the playa surface sediments: rounded concretions of microcrystals of Mg-rich calcite, confined to organic biofilms (a); evaporite halite crystal with pronounced growth zones and dissolution caverns (b); nonmineralized EPS film (c); large concretion of split sheaf-like microcrystals of Mg-calcite confined to degraded organic matter (d)

Based on the results of the microscopic studies it was shown that the terrigenous component of surface crusts of Lake Kaltyk is represented by coarse silty grains of quartz and feldspars, the predominant size is 0.06-0.09 mm. The coarse material is characterized by a significant sorting and a weak degree of roundness for individual grains. Quartz has normal extinction, weakly pronounced traces of dissolution and rare initial regeneration films. Feldspars grains are often pelitized and partially replaced by clay minerals and pelitomorphic carbonate. The carbonatization of pelitized terrigenous grains and hydrated micas presumably occurred during the formation of playa sediments. Illite and chlorite, in these deposits form the main mineral mass, forming large concretions, cementing terrigenous grains, the content of which does not exceed 20%. Authigenic minerals in the studied crusts are represented mainly by carbonates, including pelitomorphic calcite, magnesium-calcite, and aragonite. Carbonate minerals occur as rather large concretions, up to 0.2 mm in diameter, mainly developing along clay aggregates and degrading organic

matter (Figure 4c). Cubic and prismatic halite crystals, with bright growth zones and dissolution caverns, traces of recrystallization are characteristic for the surface crusts of Lake Kaltyk (Figure 4b).

An additional feature of the deposits of Lake Medvezhye is a significant admixture of *A. salina* cysts. Cysts, consisting mainly of nitrogen-containing polysaccharide (chitin), after fulfilling their direct function - preserving the body for a certain time, remain in the sediments in the form of empty membranes (Figure 5). Such shells have a high porosity and a specific surface, which allows them to actively interact with the surrounding colloidal medium. In the investigated sediments of the Medvezhye salt marsh, the shells of Artemia cysts are contained in significant volumes, sometimes up to 15-20% of the total volume of solid matter. Moreover, in drier areas located farther from the modern water line, the percentage of mineralized shells increases. The highly porous organic chitinous substrate is gradually replaced (or overgrown) with pelitomorphic microcrystalline forms of carbonates. Thus, in the presence of a large number of cysts in sediments, carbonate aggregates develop exclusively along them, without forming another kind of separation and not replacing pelitized terrigenous grains.



Figure 5. Microscopic features of *A. salina* cysts: thin section the surface crust filled with mineralized cysts (PPL) (a); SEM photo of a cyst with a preserved organic matter (b); thin sections (PPL and XPL, respectively) illustrating cyst shell replaced by pelitomorphic calcite (c and d); SEM photo of the cyst shell structure (e and f)

# Conclusion

Playas are rather common environments adjacent to large saline lakes of the Trans-Uralian region. We have studied the features of mineral composition and microstructure of the dense solid fractured crusts developed within the saline playas of Medvezhye and Kaltyk lakes. The study results showed that the surface crusts of these two sites differ in terms of proportion of terrigenous material, clay minerals, as well as the composition of evaporates and carbonates. Authigenic minerals form scattered pelitomorphic concretions, less often dense mineral nodules, often replacing pelitized fragments and organic matter. The content of terrigenous mineral components in the sediments of Lake Medvezhye is much higher than in the samples from Lake Kaltyk. Chitinous shells of Artemia cysts play a significant role in the formation of surface crusts of Lake Medvezhye. In most cases cysts are partially or completely replaced by pelitomorphic carbonates, which is clearly expressed in the sediments of Lake Medvezhye, where the *A. salina* cyst content reaches 20% of the sediment volume.

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# Spatial modeling of soil salinity using kriging interpolation techniques: A study case in the Great Hungarian Plain

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# Article Info

Abstract

Received : 21.04.20217.674 billion in 202Accepted : 18.10.2021semi-arid climates,<br/>concentrated in t<br/>physicochemical pr<br/>understanding sali<br/>resources manager<br/>model soil salinity<br/>ordinary kriging (

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The world's current task is to ensure food security for an ever-growing population of 7.674 billion in 2019. Soil degradation threatens sustainable agriculture in arid and semi-arid climates, where evaporation rates outweigh precipitation. Soluble salts concentrated in the subsoil under certain climatic conditions influence soil physicochemical properties, leading to soil fertility and biodiversity losses. Hence, understanding salinity behavior and its spatial variation are crucial for natural resources management to achieve and maintain sustainability. This study aims to model soil salinity spatial distribution using four kriging interpolation methods, i.e., ordinary kriging (OK), empirical Bayesian kriging (EBK), co-kriging (CK), and indicator kriging (IK). Two hundred twenty-two soil samples were collected for this purpose during a field campaign conducted in the Hungarian Soil Monitoring System framework in 2016. The performance of kriging methods was assessed and compared using two cross-validations, i.e., leave-one-out cross-validation (LOOCV) and the holdout method. The Pearson correlation analysis has been used to expose a significant moderate correlation between salt content and cation exchange capacity (CEC) with a correlation coefficient of 0.4 and a p-value of 0.003. Thus, the spatial relationship between soil salinity content (SSC) and CEC was integrated into the model to enhance predictions in areas where no measurements were accessible. The study demonstrated co-kriging efficiency by reducing the mean squared error (MSE) of ordinary kriging (OK) from 0.8 g/kg and 0.85 g/kg for LOOCV and the holdout cross-validation to 0.3 g/kg.

Keywords: Geostatistics, interpolation, kriging, soil salinity, spatial modeling.

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# Introduction

The accumulation of salts in the subsurface and rhizosphere, known as salinization, leads to degraded soil composition and reduced crop yield, threatening food security (Shahid et al., 2018). The global surface area affected by salinization covers approximately 831 million hectares, with 434 million hectares as sodic soils and 397 million hectares as saline soils. There are two forms of salinization based on occurrence causes. Primary salinization originates from parent material weathering, while anthropogenic actions induce secondary salinization (Uri, 2018). Extensive work using field measurements coupled with remote sensing tools, statistical analysis, geostatistics, and machine learning has been conducted to map and monitor salt-affected lands expansion over time. Many researchers have explored this topic, including Taghadosi et al. (2019), El hafyani et al. (2019), Hoa et al. (2019), Abdel-Fattah et al. (2020), and Sahbeni (2021). Geostatistical analysis generates an estimated surface from a distributed set of points using various methods, e.g., kriging, nearest neighbor, spline, inverse distance weighting (IDW), global polynomial interpolation (GPI), and conditional simulations (Emadi and Baghernejad, 2014; Sangani et al., 2019). Kriging interpolation was used by Panday et al. (2018) to determine soil chemical properties distribution over agricultural floodplain lands of the Bara district in Nepal. The study revealed a moderate spatial

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Publisher : Federation of Eurasian Soil Science Societies e-ISSN : 2147-4249 variability for pH, organic matter, nitrogen, and phosphorus. Farmers can analyze soil quality and adopt appropriate agricultural production methods based on developed maps. Abdennour et al. (2019) applied ordinary kriging (OK) and indicator kriging (IK) for salinity levels analysis in the irrigated perimeter of El-Ghrous in south-eastern Algeria. The research has determined salinity trends based on different classes and has created risk maps that decision-makers could employ in the region. Tziachris et al. (2017) compared ordinary kriging, universal kriging, and co-kriging to estimate iron content in the Kozani area. An outperformance of the co-kriging method was revealed by adding soil pH as an auxiliary variable to enhance predictions in unsampled areas. Nie et al. (2021) used kriging interpolation to study secondary salinization extent over agricultural areas in the western Jilin irrigation district, northeast China. The results showed an improvement in accuracy by 23.2% using geographically weighted regression kriging (GWRK) compared to regression kriging (RK). This provides a theoretical perspective for controlling groundwater to regulate soil salinity and prevent salinization through quantitative analysis via kriging.

The Great Hungarian Plain occupies more than 50% of Hungary's total surface, and it is contaminated by soil and wetland salinization (Mádl-Szőnyi et al., 2008). Salt content, hydraulic properties of soil, and the watertable depth influence salt-affected soils genesis in Hungary (Schofield et al., 2001), where saline soils cover 6% of its territory, making it one of the largest natural areas in Europe affected by primary salinization (Tóth et al., 2008). Thus, this expansion has inspired Hungarian scientists to study salinity behavior, origins, and restoration programs in the last decades (Tóth, 2009; Csillag et al., 1993; Tóth et al., 2002; Szatmári et al., 2020). Salinization mapping has become a valuable task for developing appropriate reclamation strategies to preserve soil quality and sustain agricultural productivity in the region. This study aims to (1) use field measurements to map salinity spatial distribution in the Great Hungarian Plain and (2) compare the predictive performance of four kriging methods, namely ordinary kriging (OK), empirical Bayesian kriging (EBK), co-kriging (CK), and indicator kriging (IK).

#### **Material and Methods**

#### Study area

The study area covers approximately 26300 km<sup>2</sup> (Figure 1), with an average altitude of 89 meters (Figure 2). Meadow chernozems and humic sandy soils dominate the landscape with an expanded agricultural land cover (Pásztor et al., 2018). The river Tisza crosses the study area, gathering tributaries from the surrounding floodplains. (Tóth et al., 2014). A warm-dry climate characterizes the study area, with an average yearly precipitation of roughly 500 mm and a mean temperature of 11°C (Hungarian Meteorological Service, 2018). May and July are the rainiest months, while January and March are the driest. Three types of deposits dominate the landscape: loess and loess-like sediments above the floodplains, silty clay in alluvial areas, and wind-blown sand on the slopes (Ronai, 1986).



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Figure 1. The study area's geographical location; (a) Satellite imagery map using ESRI basemap in ArcMap 10.3; (b) Location of sampling sites



Figure 2. Altitude map of the study area using a 30-m SRTM digital elevation model provided by the OpenTopography facility.

#### Soil Sampling and Laboratory Analysis

From mid-September to mid-October 2016, 222 soil samples were collected within the soil upper layer (30 cm) in the Hungarian Soil Monitoring System framework. The field survey is conducted in the dry season to detect the spectral properties of salts during their accumulation (Szabó and Pirkó, 2017). The Hungarian standard MSZ-08-0206/2-1978 is used to calculate soil salinity from saturated paste (MSZ 08-0206-2, 1978).

#### Semivariogram Modeling

The first step in kriging interpolation is to estimate an experimental semivariogram for the parameter to be modeled. Equation (<u>1</u>) illustrates the semivariance expression (<u>Deutsch and Journel</u>, 1998).

$$\gamma(h) = \frac{1}{2N(h)} x \sum_{i}^{N(h)} [Z(s_i) - Z(s_i + h)]^2$$
(1)

Where  $s_i$  is the location of the i<sup>th</sup> sample,

 $Z(s_i)$  is the measurement,

h is the distance between  $Z(s_i)$  and  $Z(s_i + h)$ , and

N(h) is the number of pairs  $Z(s_i)$  over the distance h.

A semivariogram depicts the estimated  $\gamma$ (h) against h values plot (Tziachris et al., 2017). Its main parameters are range, sill, partial sill, and nugget. The range (A) represents the distance to the semivariogram flattening. The sill (C) is the y-value of the model at the range, whereas the partial sill (C<sub>1</sub>) is the difference between the sill and the nugget (C<sub>0</sub>), which is the random spatial variation (Hartmann et al., 2018; Guedes et al., 2020). The spatial dependency can be measured by dividing the nugget over the sill ratio (C<sub>0</sub>/(C<sub>0</sub> + C<sub>1</sub>)). A value below 25% implies severe spatial dependence, a value within 25% and 75% range represents mild spatial dependence, while a value more than 75% represents weak dependence (Cambardella et al., 1994). Figure 3 shows the structure of a semivariogram model.



Figure 3. Typical structure of a semivariogram model (Biswas and Si, 2013)

Several model functions can be used to estimate experimental semivariograms in applied geostatistics, such as circular, spherical, exponential, gaussian, and linear (Smith, 2011). Once a model is fitted, cross-validation is conducted to evaluate the performance of the kriging method. In this context, we used samples from a random training set representing 70% of the total data to build experimental semivariogram models, while 30% of the samples were used for validation.

#### **Kriging Interpolation**

Kriging interpolation, named after Danie Krige, is a geostatistical approach for estimating unknown values based on the distance and the degree of variation between data points (Krige, 1985; Zhang, 2011; Thompson et al., 2012). A kriged estimate refers to the weighted linear combination of known values near the estimated locations. It helps produce weights resulting in optimum and unbiased estimates (Wackernagel, 2013; Xiao et al., 2016). We estimate  $\hat{Z}(s_0)$  at unknown  $s_0$  locations after measuring N data values,  $Z(s_1)$ ,  $Z(s_2)$ ,...,  $Z(s_N)$  at  $s_1$ ,  $s_2$ ,...,  $s_N$  locations.  $Z(s_i)$  represents the estimated value at the  $s_i$  location, whereas  $\lambda_i$  represents the unknown weight for the measured value at the  $s_i$  location.  $\hat{Z}(s_0)$  is computed using Equation (2).

$$\hat{Z}(s_0) = \sum_{i=1}^N \lambda_i \, \mathbf{x} \, Z(s_i) \tag{2}$$

This step aims to find the weights  $\lambda_i$  to minimize the variance Var[ $\hat{Z}(s_0) - Z(s_0)$ ].

Given that the estimator is assumed to be unbiased:

$$E[\hat{Z}(s_0) - Z(s_0)] = 0$$
(3)

 $\hat{Z}(s_0)$  is separated into two parts: a trend component  $m(s_0)$  and a residual component  $e(s_0)$ , as shown in Equation (4).

$$\hat{Z}(s_0) = m(s_0) + e(s_0)$$
 (4)

We employed ArcMap 10.3 to conduct interpolations and to evaluate the efficiency of ordinary kriging (OK), indicator kriging (IK), empirical Bayesian kriging (EBK), and co-kriging (CK) in terms of salt content prediction throughout cross-validation. Ordinary kriging is a spatial prediction approach that reduces error variance through data configuration and variogram fitting (Wackernagel, 1995). This technique has been well presented by many scholars, i.e., Negreiros et al. (2010), Hamzehpour et al. (2013), and Kiš (2016). Indicator kriging estimates a conditional cumulative distribution function at unsampled locations using spatial interpolation. It uses indicator variables to determine the likelihood of a crucial value being overridden or not at each point in the region of interest (Delbari et al., 2011; Pásztor et al., 2015). While other methods require a manual configuration to get reliable results, empirical Bayesian kriging (EBK) automates the most exigent elements to build a realistic kriging model, making it easier to obtain more accurate predictions. These parameters are estimated using a simulation algorithm. It involves little interactive modeling but produces better results for small datasets, outperforming other kriging approaches in terms of standard errors (Bhunia et al., 2016; Samsonova et al., 2017; Gribov and Krivoruchko, 2020). Cokriging is a multivariate form of ordinary kriging that uses a well-sampled variable to estimate a poorly sampled one, considering primarily significant associations (Tajgardan et al., 2010; Gräler, 2011; Babiker et al., 2018). For this purpose, we downloaded Landsat-8 OLI data acquired in August 2016. The multispectral data were atmospherically and radiometrically corrected via ENVI IDL 5.3. We computed the canopy response salinity index (CRSI) (Scudiero et al., 2017) and albedo (Silva et al., 2016). Principal component analysis (PCA) was applied to reduce the sensor spectral noise, then only the first and the second components were extracted as they contain 98% of data variance. Additionally, slope, profile curvature, and wetness index were derived from an SRTM digital elevation model (DTM), provided by OpenTopography Facility, using ArcMap 10.3. Cation Exchange Capacity (CEC) raster data were retrieved from the European Soil Database v2 Raster Library 1 km × 1 km, provided by the European Soil Data Centre (ESDAC) (Panagos et al., 2012, ESDAC, 2021). Once remotely sensed data were processed and corresponding values to samples were extracted, Pearson correlation was conducted using RStudio 1.4.1106 to outline the potential relationship between salt content values and spectral response.

#### **Cross-validation**

Two cross-validation methods were used to compare the four state-of-the-art methods. The leave-one-out cross-validation (LOOCV) method predicts using a single observation from the training set, while the left-out observations are used to train the model. Holdout cross-validation divides the data set randomly into 70% for training and 30% for validation. We calculated the mean squared error (MSE) and the mean absolute

error (MAE) for LOOCV, while the root mean square error (RMSE) and the mean squared error (MSE) for holdout cross-validation. Equations (5), (6), and (7) illustrate statistical metrics expressions.

$$RMSE = \sqrt{\sum_{1}^{n} (\hat{y}_{i} - y_{i})^{2} / n}$$
(5)

MSE = 
$$\frac{1}{n} x \sum (y_i - \hat{y}_i)^2$$
 (6)

$$MAE = \frac{1}{n} x \sum_{1}^{n} |y_{i} - \hat{y}_{i}|$$
(7)

Where n is the total number of observations,  $\hat{y}_i$  is the estimated value for the i<sup>th</sup> observation, and  $y_i$  is the actual value for the i<sup>th</sup> observation.

#### Results

#### **Exploratory Data Analysis**

Table 1 shows the key statistical parameters of field data. Salinity distribution is characterized by a mean of 0.54 g/kg and a standard deviation of 0.85 g/kg. The substantial difference between a minimum of 0 g/kg and a maximum of 8.5 g/kg indicates a wide spatial variability of this parameter.

Table 1. Descriptive statistics of salt content samples

Salt Contont (g/lig)	Min	Max	Mean	Standard Deviation
San Content (g/kg)	0	8.5	0.54	0.85

The regression analysis revealed a significant moderate association between salt content and CEC, with a correlation coefficient of 0.4 and a p-value of 0.003 (< 5%). This positive correlation was discussed by previous studies (Shainberg et al., 1980; Naseem and Bhatti, 2000). Figure 4 shows the correlation coefficients between salt content and auxiliary variables.



Figure 4. Correlation between soil salinity content and auxiliary covariates, where ssc16 refers to soil salinity content in 2016. B5, B6, and B7 are spectral bands retrieved from Landsat-8 OLI data, while PCA1 and PCA2 are the first and the second principal components of the same image. CRSI is the Canopy Response Salinity Index. Wetness index (wi), slope, and curvature are derived from a 30-m SRTM Digital Elevation Model (DEM).

#### Analysis of Semivariograms

A semivariogram describes spatial autocorrelation between measured salt content values. Features can be extracted to define each model once it is fitted across each pair of observations. Table 2 and Figure 5 summarize the findings.

Indicator kriging has a high range of 11332.56 m, followed by co-kriging with a range of 88458.18 m, respectively. The range is lower for ordinary kriging (= 5893.58 m) with a partial sill close to co-kriging (0.686 and 0.691). A high ratio of 32.1 % was found for co-kriging, revealing a moderate spatial dependence. The spatial dependence is strong for indicator kriging equals 18.5 %, whereas it is extreme for ordinary kriging with a ratio of 6.3 %.

	Model	Nugget (C <sub>0</sub> )	Partial Sill ( $C_1$ )	Range (A) (m)	Dependence (%)
OK	Gaussian	0.046	0.686	5893.58	0.063
EBK	Exponential	***	***	***	***
СК	Gaussian	0.327	0.691	88458.18	0.321
IK	Gaussian	0.018	0.078	11332.56	0.185

Table 2. The semivariogram models and their parameters for kriging methods. The spatial dependence equals  $C_0/(C_0+C_1)$ .

\*\*\* For EBK, the semivariance is rather an empirical distribution than a fixed parameter. Therefore, spatial dependence cannot be estimated.



Figure 5. Semivariograms of soil salinity content (SSC) distribution and their fitted models

For empirical Bayesian kriging, these parameters are analyzed in empirical distributions. Since several semivariograms are measured at each site, these models have neither a range nor a sill.

#### **Soil Salinity Prediction**

Based on Figure 6, predictions revealed similar patterns with minor differences for kriging methods. Areas with higher salt content predictions are condensed in the east and the center of the study area (Orange color) on lower altitudes, whereas areas with lower predictions were found west of the river Tisza, on slightly higher altitudes.

Figure 7 shows that areas with higher prediction errors (Red color) are found in the study area center due to the sparse density of sampling sites in the river Tisza vicinity. Empirical Bayesian kriging produced the lowest prediction errors with a minimal disparity in the east and the center. Yet, co-kriging (CK) yielded better results compared to ordinary kriging (OK) in terms of prediction errors distribution. Errors are overestimated by empirical Bayesian kriging compared to co-kriging with maximum values equal to 1.07 and 0.36, respectively. Table 3 illustrates the results of leave-one-out cross-validation and holdout cross-validation.

Table 3. The results of cross-validation for soil salinity prediction. The Root Mean Square Error (RMSE), Mean Squared Error (MSE), and Mean Absolute Error (MAE), expressed in g/kg, were used to compare the performance of kriging models.

CV	Error	ОК	EBK	СК	IK
Holdout Cross-Validation	RMSE	0.53	0.57	0.60	0.24
	MSE	0.85	5 0.49 0.30 0.30		0.30
	MAE	0.18	0.18	0.22	0.02
	MSE	0.80	0.42	0.30	0.30



Author: G. SAIIBENI | Data Source: RISSAC, 2020

Figure 6. Prediction maps for salt content (g/kg) spatial distribution using kriging interpolation methods, (a) OK; (b) EBK; (c) CK; (d) IK



Figure 7. Prediction error maps of salt content (g/kg) spatial distribution using kriging interpolation methods, (a) OK; (b) EBK; (c) CK; (d) IK

Co-kriging and indicator kriging produced the lowest MSE (= 0.3 g/kg) for the holdout cross-validation and leave-one-out cross-validation. Meanwhile, ordinary kriging has the highest MSE for holdout cross-validation and LOOCV, equal to 0.53 g/kg and 0.8 g/kg, respectively. For RMSE, indicator kriging has the lowest value (= 0.24 g/kg), followed by ordinary kriging (= 0.53 g/kg), empirical Bayesian kriging (= 0.57 g/kg), and co-kriging (= 0.6 g/kg). For MAE, indicator kriging has the lowest value, equal to 0.02 g/kg, followed by ordinary kriging, and co-kriging. Overall, kriging methods have close RMSE and MAE values except for indicator kriging as it produces probabilities rather than numeric values.

Co-kriging performed well in terms of MSE reduction for both cross-validation stages compared to ordinary kriging. In contrast, empirical Bayesian kriging and indicator kriging showed better performance in terms of MAE, revealing a remarkable potential to produce unbiased predictions. The superiority of co-kriging was expected due to its hybrid structure, conditionally with a strong correlation between variables as the spatial distribution of one parameter is exploited to estimate the behavior of the second one. Despite the significant improvement of predictions using co-kriging, the correlation between salt content and CEC is moderate (40%), which limited the model from outperforming other methods in terms of RMSE. This issue can be investigated in future studies.

# Discussion

This study demonstrates geostatistics efficiency in mapping salinity distribution with significant accuracy, supported by the studies of Gallichand et al. (1992) and Pulatov et al. (2020). Benslama et al. (2020) interpolated the electrical conductivity (EC) using inverse distance weighting (IDW) and ordinary Kriging (OK). Interpolation methods performed well in soil salinity mapping, with mean error (ME) and root mean square error (RMSE) of -0.003 dS/m and 0.145 dS/m, respectively. In the same context, Nezami and Alipour (2012) examined the potential of interpolation methods in salinity mapping across the Qazvin Plain, i.e., ordinary kriging (OK), co-kriging (CK), spline, and inverse distance weighted (IDW). The results revealed that co-kriging performed well due to the integration of clay content (%), with a root mean square error (RMSE) equal to 108 dS/m<sup>2</sup>. This agrees with our findings regarding the outperformance of co-kriging in modeling salinity distribution with a prediction error ranges between 0.04 g/kg and 0.36 g/kg. The cokriging model of Abdennour et al. (2020) yielded a root mean square error (RMSE) of 0.92 dS/m and a mean error (ME) of 0.004 dS/m, revealing an accuracy enhancement by adding saturation index (SI) of gypsum and SO<sub>4</sub><sup>2-</sup>, NDVI, and terrain parameters. Using CEC as an auxiliary covariate, our cokriging model produced better predictions in terms of soil salinity modeling with a root mean squared error (RMSE) equals 0.6 g/kg and a mean squared error (MSE) equals 0.3 g/kg. However, more improvement can be made by including variables with stronger associations rather than the cation exchange capacity (CEC). Triantafilis et al. (2001) compared four kriging methods, i.e., ordinary kriging, regression kriging (RK), dimensional kriging (DK), and co-kriging (CK), using electromagnetic induction data across an irrigated cotton in the Namoi valley (Australia). Despite the superiority of regression kriging in precision and estimation bias, co-kriging produced more accurate predictions, supporting our findings. The ordinary kriging method performed well with an RMSE equal to 0.53 g/kg, agreeing with Nawar et al. (2011) study that revealed ordinary kriging potential based on a spherical semivariogram for mapping salinity distribution in El-Tina Plain (Egypt). Similarly, Zheng et al. (2009) predicted electrical conductivity (EC) variation in the west margin of Taklamakan desert using ordinary kriging (OK). The model produced an efficiency factor (E) of 0.7793 mS/cm and a prediction ratio to deviation (RPD) of 0.39 mS/cm. The study agrees with our findings regarding ordinary kriging efficiency as a promising alternative to spatially map salinity with acceptable accuracy.

An interpretation of prediction maps showed that higher salinity predictions are concentrated around lower altitudes around the river Tisza and Hortobágy National Park, whereas lower salinity predictions are frequent in slightly higher altitudes. Topography and climate play a crucial role in salt accumulation and movement within the subsoil, as discussed by many scholars (Schofield et al., 2001; Ivushkin et al., 2017; Sahbeni, 2021). Thus, it is recommended to adopt a practical field sampling approach that preserves the continuity and the representativeness principles. To produce more accurate results, distance between sites, sampling density, geologic formations, and topographic parameters of the study area must be considered.

# Conclusion

The current study investigates kriging potential in salinity prediction. Implementing a suitable method that minimizes errors and predicts salt content accordingly offers a quick and affordable approach to monitoring salinity variation and preventing its expansion. In this context, co-kriging performed well in terms of

prediction error distribution that varies between 0.04 g/kg and 0.36 g/kg using cation exchange capacity (CEC) as an auxiliary covariate. Stronger associations with soil salinity in the vicinity of unsampled or poorly sampled sites can improve co-kriging performance and reduce prediction error. On the other hand, indicator kriging outperformed other methods in terms of MSE, MAE, and RMSE, with values of 0.3 g/kg, 0.02 g/kg, and 0.24 g/kg for LOOCV and holdout cross-validation, respectively. We have found that more than 10% of the study area surpassed the 1g/kg threshold limit, which might require immediate intervention to validate this scenario. Overall, salinity distribution maps can assist in boosting awareness of reclamation programs for sustainable agriculture and implementing feasible planning strategies. Nevertheless, the sample size and the non-uniform distribution of samples affected prediction errors by overestimating or underestimating in areas where no or few samples were taken. Further research must be conducted on larger scales (agricultural lands/farms scale) to validate the applicability of the proposed approach.

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# Properties, geochemical composition, and fertility of highly weathered soils in Central Philippines

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## Abstract

Highly weathered soils are widepsread in the humid tropics. These soils are deep, clayey, and reddish and contain scant amounts of nutrients like Si due to excessive leaching, heavy desilication-aluminization, and erosion owing to their slope gradient and position. However, few investigations have been published in terms of their nature, characteristics, and nutrient status, especially for the geologically young Philippine islands. This study assessed the properties, geochemical composition, and fertility of deep and highly weathered soils developed from various parent materials in Central Philippines (Leyte and Samar). Sampling covered the entire soil profiles, including the lower portions, that are usually neglected in common soil characterization studies. Among the soil profiles, only profiles 3 and 8 have developed from non-uniform or heterogeneous parent materials. Findings likewise revealed heavy losses of K<sub>2</sub>O, CaO, MgO, and Na<sub>2</sub>O from the highly soil profiles evaluated. The amount and profile distribution of K<sub>2</sub>O and CaO is below 0.5% in the entire profile of most soils. On the other hand, there is apparent enrichment of Al<sub>2</sub>O<sub>3</sub>,  $Fe_2O_{3}$ , and to a lesser extent SiO<sub>2</sub> in the highly weathered soil profiles, thus supporting the residua hypothesis. In terms of morpho-physical characteristics, the soils have deep solum, reddish color, subangular structure, friable moist consistence, and sticky and plastic wet consistence which are all related to the highly weathered nature of the soils. They also generally have low bulk density and higher porosity due to the iron oxides aggregation effect. The strong acidity (pH <5) and negative delta pH values revealed that the soil colloids possess a negative net charge. Nutrient status also showed low contents of organic matter, total N, available P, and exchangeable bases. Majority of the deep and highly weathered soils evaluated have possibly developed from homogenous parent materials. The soils are classified as Hapludox, Hapludult, and Paleudult.

Keywords: Geochemical composition, highly weathered soils, nutrient status.

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# Introduction

Weathering is the disintegration of rocks on the earth's surface as a result of physical, chemical, and biological processes. According to Strakhov (1967), although weathering is a fundamental characteristic of humid regions, the degree of its development differs depending on the combination of temperature and moisture, the introduction of organic material, and the relief of the region, the latter being greatly influenced by tectonic uplift. He further explained that the maximum intensity of weathering is attained in the moist tropics resulting in the thickest weathered residue with a characteristic geochemical composition (Strakhov, 1967). As first demonstrated by V.V. Dokuchaev, the products from rock weathering are subjected to various factors such as climate, parent rock, organisms, topography, and time all of which determine the nature of the soil that develops (Jenny, 1941; Barshad, 1964; Kyuma, 2021).

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Publisher : Federation of Eurasian Soil Science Societies e-ISSN : 2147-4249 Highly weathered soils are prevalent in the wet tropics, often derived from Precambrian rocks, developed from pre-weathered parent materials (Stoops, 2003), and have undergone prolonged and intense weathering under the net leaching environment, specifically humid tropics (Mohr et al., 1972; Sanchez, 1976).

Humid tropics are estimated to be covered by 70% of strongly weathered soils (Dudal, 2003). These soils are deep, clayey, and reddish and mostly contain low amounts of nutrients like Si due to excessive leaching, heavy desilication-aluminization, and erosion due to their slope gradient and position. Strongly weathered soils with low contents of weatherable minerals and with clays consist mainly of kaolinite and oxides of iron and aluminum.

Chesworth (1973a) hypothesized that there is a tendency to simplify weathering products with time towards what he calls a "residua system" composed of  $SiO_2$ ,  $Al_2O_3$ ,  $Fe_2O_3$ , and  $H_2O$ . This implies that highly weathered soils would have comparable geochemistry even if derived from different parent materials. The typical minerals in weathering residue are quartz, a hydrous form of silica (opal and chalcedony), gibbsite, boehmite, goethite, hematite, kaolinite, and halloysite.

On the other hand, accumulation of Titanium (Ti) and Zircon (Zr) minerals are higher in tropical regions than in temperate with high anion and cation adsorption and pH-dependent charged as observed in titaniferous Oxisols from Malaysia (Fitzpatrick and Chittleborough, 2002).

Recent studies on highly weathered soils in Central Philippines determined the properties, geochemical composition, and fertility in the lower portions of the deep soil properties (Asio, 1996; Navarrete et al., 2007; Piamonte et al., 2014). Evaluating these parameters will contribute to a better understanding of the genesis of the soil, especially from the Philippines. Thus, this study was conducted to assess the properties, geochemical composition, and fertility of deep and highly weathered soils developed from various parent materials in Central Philippines (Leyte and Samar) of the entire soil profile, including the lower portions, which are usually ignored in common soil characterization studies.

# Material and Methods

#### **Site Selection and Characteristics**

Eight soil profiles at various elevations and physiographic positions were selected from several parts of Central Philippines. Specifically, places include Leyte (Baybay and Silago) and Samar (Salcedo, Hernani, and Hinabangan) (Figure 1). The climate is classified as Af (tropical wet climate) in the Köppen climatic classification and as Type II and Type IV in the Coronas climatic classification of the Philippines (Coronas, 1920). Rainfall is evenly distributed throughout the year, with an average annual rainfall of  $\sim$  2800mm. Diverse plant species dominated the areas. The soil moisture regime is udic, which implies that the water is available year-round. In addition, the soil temperature regime is hyperthermic, with an annual average temperature of above 22°C, and it does not fluctuate above 5°C (Soil Survey Staff, 1999). Table 1 presents the detailed characteristics of the sampling sites.

#### Soil Profile Description and Sampling

Preference was set to recent road cuts to get samples from deeper soil layers. Soil profile descriptions were done following the standard procedure of FAO Guidelines for Soil Description (Jahn et al., 2006). Soil samples were taken from each horizon of every soil profile quantitatively by taking three (3) continuous and even slices from the uppermost horizon down to the lowest and were mixed thoroughly (Schlichting et al., 1995). Wider soil slices were collected in thin horizons to ensure that the sample volume is approximately equal to those from the thicker horizons. For the purpose of comparison, sampling was done using a uniform measurement of (0-20 cm, 20-40 cm, 40-70 cm, 700-100 cm, 100-130 cm, 130-160 cm, 160-190 cm 190-210 cm, 210-240 cm, 240-270 cm, 270-300 cm, 300-330 cm, 330-360 cm and 360-400 cm). All soil samples were placed in a properly labeled plastic and brought to the Department of Soil Science Visayas State University for processing. Samples were unfettered from rocks and leaves, air dried, pulverized, and sieved in different wire mesh sizes to determine physical and chemical properties.

#### **Laboratory Analyses**

The total elemental composition was analyzed by X-ray fluorescence analytical microscope (HORIBA XGT-7200). Bulk density (g/cm<sup>-3</sup>) was determined using the paraffin-clod method (Blake and Hartge, 1986) and percent (%) porosity was computed from the bulk density value and an assumed particle density value of 2.65 g/cm<sup>3</sup> (ISRIC, 1995). Particle size distribution was determined using a pipette after pretreatment with H<sub>2</sub>O<sub>2</sub> to oxidize organic matter (ISRIC, 1995). Water holding capacity (WHC) and field capacity (FC) were

determined using the gravimetric method (Klute, 1986). Soil pH (1:25 H<sub>2</sub>O) and 0.01 M KCl were measured using pH meter, and total nitrogen (N) was determined following the Micro-Kjeldahl method (ISRIC, 1995). Percent (%) organic matter (OM) was measured by the modified Walkley-Black method (Nelson and Sommers, 1996). Available P was obtained following the Bray P2 (ISRIC, 1995). The exchangeable bases (cmolc kg<sup>-1</sup>) were quantified following the ammonium acetate method (Jones, 2001), and quantified using Agilent 4200 (microwave plasma-atomic emission spectrometry).



Figure 1. Study sites in Leyte and Samar, Philippines

Γable 1. Site characteristics of the	e sampling sites of o	deep and highly we	athered soils in Leyte and Samar
	1 0	1 0 7	

Site Characteristics				PROF	ILE NO.			
	1	2	3	4	5	6	7	8
Location	Naparaan,	Naparaan,	Hernani, Samar	Bagacay,	Mt. Pangasugan	Tubod, Silago, So.	Imelda, Silago So.	Makinhas,
	Salcedo E. Samar	Salcedo E. Samar		Hinabangan Samar	Baybay Leyte	Leyte	Leyte	Baybay Leyte
Coordinates	N10°08'941"	N11°09'676"	N11°16'895"	N11°49'675"	N10°44'901"	N10°32.323'	N10°33'734"	N10°38'890"
	E125°39'313"	E125°037'452"	E125°33'588"	E125°12'415"	E124°48'206"	E125°8.777"	E125°05'48.3"	E124°51'621"
Elevation	65 m asl	100 m asl	26 m asl	251 m asl	129 m asl	156 m asl	369 m asl	82 m asl
Slope Formation	Backslope	Upper Backslope	Shoulder	Backslope	Upper backslope	Upper backslope	Summit	Summit
Slope Gradient	Sloping	Sloping	Sloping	Sloping	Sloping	Sloping	Nearly level	Gently sloping
Parent Material	Serpentinized	Serpentinized	Ophiolite	Slate	Andesite-Basalt	Basaltic-andesitic	Basaltic-	Andesite
	Ultrabasic Rock	Ultrabasic Rock				volcanics	andesitic	
							volcanics	
Soil Moisture Regime	Udic	Udic	Udic	Udic	Udic	Udic	Udic	Udic
Soil Temperature	Isohyperthermic	Isohypertherm	Isohypertherm	Isohyperthermic	Isohyperthermic	Isohyperthermic	Isohyperthermic	Isohyperthermic
		ic	ic					
Erosion	Slight Water	Slight Water	Moderate	Slight Water	Slight Water	No evidence	No evidence	No evidence
	Erosion	Erosion		Erosion	Erosion			
Rock outcrops	Few	Few	Common	Few	Few	Very few	Very few	Very few
Drainage	Well-drained	Well-drained	Well-drained	Well-drained	Well-drained	Poorly drained	Well-drained	Well-drained
Land-use	Dipterocarp	Dipterocarp	Degraded land	Forest	Secondary forest	Agricultural	Secondary Forest	Secondary Forest
	forest	forest						
Vegetation	Deciduous woods	Deciduous	Pteridium	Deciduous	Dipterocarps,	Ferns, cogon	Ferns, cogon,	Coconut, grass,
		woods	aquilinum	woods	Grasses		taro, goatweed	ferns

# Results

#### **Uniformity of Parent Material**

The depth function of  $TiO_2$  and  $ZrO_2$  defined the characteristic of parent materials (Figure 2). Soil profiles (3, 5, 6, and 8) showed considerable variations of  $TiO_2$  and  $ZrO_2$  with depth, indicating possible heterogeneity of the parent materials. Conversely, for the depth function of  $ZrO_2$ , only profiles 3 and 8 show considerable variations with depth. From these two findings, it is most likely that profiles 3 and 8 have developed from non-uniform or heterogeneous parent materials.



Figure 2. Depth functions of TiO2 and ZrO2 in deep and highly weathered soils in Leyte and Samar

#### **Composition of the Weathered Residue**

The depth function of SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, and Fe<sub>2</sub>O<sub>3</sub> demonstrated the composition of the weathered residue (Figure 3). Most of the soils except profiles 1 and 3 have close to 40% SiO<sub>2</sub> in their profiles which generally appeared uniform with soil depth. This implies that weathering of these soils has been intensive down to a few meters' depths. For Al<sub>2</sub>O<sub>3</sub>, most soils have a uniform distribution with depth and contain considerable amounts (between 15 and 35%) because of the intensive weathering process. However, Fe<sub>2</sub>O<sub>3</sub> findings reveal high amounts ranging from 30 to 50%.



Figure 3. Depth functions of SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, and Fe<sub>2</sub>O<sub>3</sub> in deep and highly weathered soils in Leyte and Samar

#### Amounts of Basic Elements (K<sub>2</sub>O, CaO, MgO, and Na<sub>2</sub>O)

Findings revealed generally heavy losses of  $K_2O$ , CaO, MgO, and Na<sub>2</sub>O from the highly soil profiles evaluated (Figure 4). The amount and profile distribution of  $K_2O$  and CaO is below 0.5% in the entire profile of most soils. Profiles 1, 2, and 8 show a slight tendency for  $K_2O$  to increase with soil depth, suggesting the possible contribution of deep rock weathering. On the other hand, MgO and Na<sub>2</sub>O are much higher in all the soils than CaO and  $K_2O$ . This suggests that CaO and  $K_2O$  are more mobile in the weathering environment compared to MgO and Na<sub>2</sub>O.





#### Other Elements (CuO, ZnO, PbO, NiO, CrO, and SO<sub>2</sub>)

The amounts of CuO, ZnO, PbO, NiO, CrO, and  $SO_2$  in the soil profiles were very small except for profiles 1 and 3, revealing considerable amounts of CrO. Also, profile 3 had more NiO compared to other soil profiles (Figure 5).

#### **Soil Morpho-Physical Properties**

Table 2 presents the morphological properties of the highly weathered soils evaluated. Soil profile 1 has a clay texture and a reddish brown surface horizon which turns to red in the subsurface. Soil profile 2 is a silty clay and very deep soil with a color ranging from brown to orange in the subsoil. Soil profile 3 is a sandy clay soil varying in color from yellowish brown to brown. All three profiles showed no rock fragments in their profile. They have subangular blocky structure, friable moist consistence but sticky and plastic wet consistence. Soil profiles 4 and 5 are clayey deep soils ranging in color from bright reddishbrown to red. They have subangular blocky structure, friable moist consistence and sticky and plastic wet consistence. Soil profiles 6, 7, and 8 have closely similar soil morphology especially in terms of color, structure, and consistence. Results also revealed that the bulk density values of the soils are below 1.0 g/cm<sup>3</sup>, porosity values range from 60 to 80%, and water holding capacity is high 75-95% (Figure 7). Also, the soil profiles 1, 2, and 5 had the highest clay contents (Figure 8).



Figure 5. Depth functions of CuO, ZnO, PbO, NiO, CrO, and SO<sub>2</sub> in deep and highly weathered soils in Leyte and Samar **Soil Chemical Properties** 

Most of the highly weathered soils evaluated are strongly acidic. Moreover, they have a higher pH in  $H_2O$  than pH in KCl (Figure 9). They also have low organic matter contents and P availability (Figure 10). Except for the surface horizons of soil profiles 4 and 6, all other soil profiles have lower than 3% organic matter content, which decreased to below 1% in the lower portions. All soil profiles also revealed low exchangeable base contents (Supplementary Table 1).

							Consi	stence <sup>F</sup>	
Horizon <sup>A</sup>	Depth	Boundary <sup>B</sup>	Color (Moist)	Texture <sup>c</sup>	Rock fragments <sup>D</sup>	Structure <sup>E</sup>	Wet	Moist	Roots <sup>G</sup>
	(cm)								
			I	Profile 1					
Ah	0-20	CS	2.5YR 4/6 reddish brown	SiC	n	3fg	fr	spl&st	vff
Bo1	20-40	CS	2.5YR 4/6 reddish brown	С	n	3abk	fr	spl&sst	fm
Bo2	40-70	CS	2.5 YR 4/6 reddish brown	C	n	3SDK	vfr	pl&st	fm
B03 Bo4	/0-100	cs	2.5 YR 3/6 Drown	C	n	3SDK 2cblz	vir	plast	CI
Bo5	130-160	ds	10R 4/6 red	C	n	3sbk	vfr	nl&st	
Bob	160-190	ds	10R 4/8 red	C	n	3sbk	vfr	pl&st	-
Bo7	190 below	ds	10R 4/6 red	C	n	3sbk	vfr	pl&st	-
			, i i i i i i i i i i i i i i i i i i i	Profile 2				•	
Ah	0-20	ds	10YR 4/6 brown	SiC	n	3fg	1	sst&spl	fm
AB	20-40	ds	7.5 YR 5/6 bright brown	SiC	n	3fg	fr	st&pl	ff
Bo1	40-70	ds	7.5 YR 5/6 bright brown	SiC	n	3sbk	fr	sst&spl	ff
B02	/0-100	ds	7.5 YR 6/8 orange	SIC	n	3SDK	fr	st&pl	fm
B03 Bo4	120 160	ds	7.5 YR 5/6 bright brown 7 5 YR 5/6 bright brown	SIC	n	3SDK 2cblz	II fr	st&pl	fm
Bo5	160-190	ds	7.5 VR 4/6 orange	SiC	n	3shk	n fr	st&pl	fm
Bob	190-210	ds	7.5 YR 5/6 bright brown	SiC	n	3sbk	fr	st&pl	fm
Bo7	210-240	ds	7.5 YR 6/8 orange	SiC	n	3sbk	fr	sst&spl	fm
Bo8	240-270	ds	7.5 YR 6/8 orange	SiC	n	3sbk	fr	sst&spl	fm
Bo9	270-300	ds	7.5 YR 6/8 orange	SiC	n	3sbk	fr	sst&spl	fm
Bo10	300-330	ds	7.5 YR 6/8 orange	SiC	n	3sbk	fr	st&spl	fm
Bo11	330-360	ds	7.5 YR 6/8 orange	SiC	n	3sbk	fr	st&spl	fm
Bo12	360 below	ds	7.5 YR 6/8 orange	SiC	n	3sbk	fr	st&spl	fm
	0.00			Profile 3		26	<i>c</i>	.0.1	
Ah	0-20	ds	10YR 5/6 yellowish brown	SC	n	2fg	fr	st&spl	cm
Bo1	20-40	ds	10YR 4/6 brown	SC	n	2fsbk	fr	st&spl	ff&vfm
Bo2	40-70	ds	10YR 4/6 brown	SC	n	2fsbk	fr	st&spl	ff&vfm
Bo3	70-100	ds	10YR 4/6 brown	SC	n	2fsbk	fr	st&spl	ff&vfm
Bo4	100-130	ds	10YR 4/6 brown	SC	n	2fsbk	fr	st&spl	ff&vfm
Bo5	130-160	ds	10YR 4/6 brown	SC	n	2fsbk	fr	st&spl	ff&vfm
Bo6	160-190	ds	10YR 4/6 brown	SC	n	2fsbk	fr	st&spl	ff&vfm
BC1	190-210	ds	10YR 4/6 brown	SC	n	2fsbk	fr	st&spl	ff&vfm
BC2	210-240	ds	10YR 4/6 brown	SC	n	2fsbk	fr	st&spl	ff&vfm
BC3	240 below	ds	10YR 4/6 brown	SC	n	2fsbk	fr	st&spl	ff&vfm
				Des Class					
				Profile 4					
Ah	0-20	CS	5YR 5/6 bright reddish brown	SiCl	n	3fg	fi	st&spl	cf
BA	20-40	CS	2.5YR 5/6 bright brown	SiCl	n	3sbk	fi	sst&spl	ff
Bt1	40-70	CS	2.5 YR 6/8 bright reddish brow	n HC	n	3sbk	vf	vst&vpl	vf
Bt2	70-100	d	10R 4/6 red	HC	n	3sbk	vf	vst&vpl	vf
Bt3	100-130	d	10YR 4/8 brown	HC	n	3sbk	vf	vst&vpl	vf
Bt4	130-160	d	10YR 4/8 brown	HC	n	3sbk	vf	vst&vpl	vf
Bt5	160-190	d	10B 4/6 red	нс	n	3shk	vf	vst&vnl	vf
Dt5	100 210	d	10R 4/8 rod	нс	n	2 chlr	ví	votevpl	vi
DLO Dt7	190-210	u	10R 4/6 red	IIC	11	2-bl	VI	vstævpi	VI C
Bt/	210-240	d	10R 4/6 red	HC	n	3SDK	vf	vst&vpl	vf
Bt8	240 below	d	10R 4/6 red	HC	n	3sbk	vf	vst&vpl	vf
				Profile 5					
Ah	0-20	CS	7.5YR 3/4 bright brown	С	n	1fsbk	fr	st, pl	cf
Bw1	20-40	CS	5YR 5/8 bright reddish	С	f	3sbk	fr	st, pl	cff&ff
Bw2	40-70	CS	5YR 5/8 bright reddish	С	f	3sbk	fr	st. pl	cff&ff
Bt1	70-100	d	7.5YR 4/6 brown	C	f	3shk	fr	st. nl	cff&ff
Bt2	100-120	ch	5VR 4/6 reddich brown	C	f	Schle	fr	st pl	cff9.ff
Dt2	120 1/0	c.b.	STR +/ G requisit Drown	C C	1 C	2 abl	11 6	st, pi	
BI3	130-160	CD	51K 4/6 bright readish brown	L	1	SSDK	fr	st, pi	cli&ff
BC	160-190	d	5YK 4/6 bright reddish brown	С	t	3sbk	fr	st, pl	cff&ff
CB1	190-210	cb	2.5 Y 5/6 bright brown	С	f	3sbk	fr	st, pl	cff&ff
CB2	210-240	cb	2.5 Y 5/6 bright brown	С	f	3sbk	fr	st, pl	cff&ff
CB3	240-270	cb	2.5 Y 5/6 bright brown	С	f	3sbk	fr	st, pl	cff&ff
Cw	270 below	Dominated l	oy saprolite, appears massive but	t can be easily b	oroken by shovel			27,027,67	

# Table 2. Morphological characteristics of deep and highly weathered soils in Central Philippines

#### Table 2 (continue)

							<u>Consistence<sup>F</sup></u>		
Horizon <sup>A</sup>	Depth (cm)	Boundary <sup>B</sup>	Color (Moist)	Texture <sup>c</sup>	Rock fragments <sup>D</sup>	Structure <sup>E</sup>	Wet M	oist Ro	ots <sup>G</sup>
	(em)								
				Profile 6					
Ah	0-20	as	10YR 3/4 (dark yellowish	CL	n	1fsbk	fr	st & pl	fm
			brown)					5	
Bw	20-40	as	10YR 5/8 (yellowish brown)	С	n	1fsbk	fi	st & pl	cf
Bt1	40-70	gs	5YR 5/8 (yellowish red)	С	n	2fsbk	vfi	vst & vpl	cf
Bt2	70-100	gs	5YR 5/8 (yellowish red)	С	n	2fsbk	vfi	vst & vpl	vff
Bt3	100-130	gs	5YR 5/8 (yellowish red)	С	n	2fsbk	vfi	vst & vpl	-
Bt4	130-160	gs	5YR 5/8 (vellowish red)	С	n	2fsbk	vfi	vst & vpl	-
BC1	160-190	gs	5YR 5/8 (yellowish red)	С	С	2fsbk	vfi	vst & vpl	-
BC2	190-210	gs	5YR 5/8 (yellowish red)	С	С	2fsbk	vfi	vst & vpl	-
BC3	210-240	gs	5YR 5/8 (vellowish red)	С	с	2fsbk	vfi	vst & vpl	-
BC4	240-270	gs	5YR 5/8 (vellowish red)	С	с	2fsbk	vfi	vst & vpl	-
BC5	270 below	gs	5YR 5/8 (yellowish red)	С	с	2fsbk	vfi	vst & vpl	-
		0		Profile 7					
Ah	0-20	CS	5YR 4/6 (vellowish red)	CL.	n	1fshk	fr	sst & snl	cf
Bw	20-40	as as	5YR 4/6 (vellowish red)	CL.	n	1fshk	fr	sst & spl	cf
Bt1	40-70	53 0W	5YR 4/6 (vellowish red)	C	n	2mshk	fi	st & nl	cf
Bt2	70-100	gw	5YR 5/6 (vellowish red)	C	n	1fshk	fr	vst & vnl	vff
Bt3	100-130	gw	5YR 4/6 (vellowish red)	C	n	1fsbk	fr	vst & vpl	vff
BC1	130-160	CS CS	5YR 4/6 (vellowish red)	C	r C	1fsbk	fr	vst & vpl	vff
BC2	160-190	CS CS	5YR 4/6 (vellowish red)	Č	c	1fshk	fr	vst & vnl	vff
BC3	190-210	CS	5YR 4/6 (vellowish red)	C	c	1fsbk	fr	vst & vpl	vff
BC4	210 below	CS	5YR 4/6 (vellowish red)	Č	c	1fsbk	fr	vst & vpl	vff
				Profile 8			185		
Ah	0-20	dw	5YR 4/4 reddish brown	SiC	f	3fsbk	fr	Sst&spl	cm
Bt1	20-40	dw	5YR 5/6 vellowish red	С	f	3fsbk	fr	Vst& pl	cm
Bt2	40-70	dw	5YR 5/6 vellowish red	SL	f	2fsbk	fr	St&pl	cm
Bt3	70-100	dw	5YR 5/8 yellowish red	SL	f	2fsbk	fr	Vst&pl	ff
Bt4	100-130	ds	5YR 5/8 yellowish red	L	f	3msbk	fr	Sstk&spl	ff
BC1	130-160	ds	2.5YR 4/8 red	L	с	3msbk	fr	Sstk&spl	ff
BC2	160-190	ds	2.5YR 4/8 red	SiC	С	3msbk	fr	Sstk&spl	-
BC3	190-210	dw	2.5YR 4/8 red	SiC	с	1fsbk	fr	Sstk&spl	-
BC4	210 below	dw	2.5YR 4/8 red	SiC	С	1fsbk	fr	Sstk&spl	vfm

A -Based on IUSS Working Group WRB (2015)

B -ds, diffuse smooth; dw, diffuse wavy; cw, clear and wavy; cs, clear and smooth

C -SC, Sandy clay; SL, sandy loam; SCL, Sandy clay loam; C, clay; L, loam

D -1, weak; 2, moderate; 3, strong; vf, very fine; f, fine; m, medium; sbk, sub-angular blocky; abk, angular blocky; g, granular

E -n, no rock fragments; f, few; c, common

F -fi, firm; vfr, very friable; fr, friable; nst, non-sticky; sst, slightly sticky; st, sticky; vst, very sticky; npl, non-plastic; spl, slightly plastic; pl, plastic; pvp, plastic to very plastic; vpl, very plastic

G-vff, very few fine; ff, few fine; cf, common fine; fm, few medium; vfm, very few medium; cm, common medium; fm, fine medium

# Discussion

#### **Uniformity of Parent Material**

Blume (1963) and Barshad (1964) explained that the first task in the evaluation of soil profile development is the establishment of the uniformity of the parent material from which the soil has developed. Stahr (1979) and Alaily (1984) reported that indications of heterogeneity of parent material are inferred from depth functions and distribution of morphological, physical, chemical, and mineralogical properties which cannot be explained by pedogenesis. Barshad (1964) recommended the use of stable minerals or elements such as  $TiO_2$  and  $ZrO_2$  although he emphasized that it is not necessary that all stable elements should have a uniform distribution in the soil profile. Asio (1996) found that  $Al_2O_3$ ,  $TiO_2$  and  $ZrO_2$  were the most suitable for establishing parent material uniformity of soils developed from volcanic rocks in Leyte, Philippines. In this present study, the depth function of  $ZrO_2$  showed that only two of the eight highly weathered soil profiles have developed from non-uniform parent materials and thus it is contrary to the widespread notion that highly weathered tropical soils are polygenetic and developed from heterogenous pre-weathered parent materials (Stolbovoy, 1992; Mohr et al., 1972; Birkeland, 1984).



Figure 6. Eight soil profiles showing deep and highly weathered soils in Salcedo, Hernani and Bagacay Samar (Soil profile1-4), Baybay and Silago Southern Leyte (Soil profile 4-8)



Figure 7. Depth function of bulk density, percent (%) porosity and water holding capacity in deep and highly weathered soils in Leyte and Samar



Figure 8. Particle Size Distribution of deep and highly weathered soils in Leyte and Samar



Figure 9. Depth functions of pH values (H<sub>2</sub>O and KCl) in deep and highly weathered soils in Leyte and Samar

#### **Composition of the Weathered Residue**

Chesworth (1973a) proposed the residua system hypothesis to explain the weathering of silicate rocks. He specified that the weathered products (residua system) would move towards  $SiO_2 - Al_2O_3 - Fe_2O_3-H_2O$  system. This hypothesis helps explain the findings of this study for the apparent enrichment of  $Al_2O_3$ ,  $Fe_2O_3$ , and to a lesser extent  $SiO_2$  in the highly weathered soil profiles evaluated. Mohr et al. (1972) reported several profiles of highly weathered soils from Indonesia that showed generally comparable levels of  $SiO_2$ ,  $Al_2O_3$ , and  $Fe_2O_3$  with the soils that we have investigated. Tsozue and Yongue-Fouateu (2017) observed the accumulation of aluminum and iron oxides during the weathering of micaschist in the rainforest of Cameroon. Asio and Jahn (2007) likewise reported  $SiO_2$ ,  $Al_2O_3$ , and  $Fe_2O_3$  enrichment in highly weathered soils from basalt in Leyte, Philippines.

According to Chesworth (1973b), the chemical imprint left upon soil by its parent material diminishes with time. This means that the effect of parent material on derived soil is an inverse function of time. This suggests that weathering of different rocks under identical humid tropical conditions could produce soils of closely similar geochemical compositions.

This explains the findings of the present study in which different parent materials produced closely related soils classified as Oxisols and Ultisols. Mohr et al. (1972) showed examples of highly weathered humid tropical soils having similar mineralogical composition in the clay fraction although they were derived from different parent materials.



Figure 10. Depth functions of organic matter, total nitrogen (%) and available P of deep and highly weathered soils in Leyte and Samar.

#### Amounts of Basic Elements (K<sub>2</sub>O, CaO, MgO and Na<sub>2</sub>O)

Mohr et al. (1972) reported extensive data from various parts of the humid tropics showing heavy losses of K, Ca, Mg, and Na during intense weathering. Asio (1996) and Asio and Jahn (2007) observed that intensive chemical weathering resulted in substantial loss of elements, particularly Ca, Mg, K and Na, during basalt weathering into the deep, clayey, and acidic soil in the study site (Leyte, Philippines). Such heavy losses are due to the high mobility of these basic cations in the weathering environment. According to the Polynov's ion mobility series (Polynov, 1937), Ca is the most mobile followed by Na, then by Mg and K. Studies have shown that the sequence slightly varies depending on environmental conditions. Middelburg et al. (1988) suggested that the Polynov's ion mobility series is applicable only on a global scale but not on smaller scales where the mineralogy of the rock is the predominant factor controlling the mobility of major elements.

#### **Soil Morpho-Physical Properties**

The morpho-physical properties of the soils particularly in terms of type of horizons, soil depth, color, texture, structure, and consistence are typical of highly weathered tropical soils (e.g. Juo and Franzluebbers, 2003). The deep solum, reddish color, subangular structure, friable moist consistence, and sticky and plastic wet consistence are all related to the highly weathered nature of the soils (Mohr et al., 1972; Sanchez, 1976; Jahn and Asio, 2006). As was elucidated by Strakhov (1967), the high temperature and high rainfall in the humid tropics enhance the intense weathering resulting in deep soils. The brown and reddish color of highly weathered tropical soils is due to the considerable amounts of iron oxides produced by the intense weathering. The friable consistence is attributed to the excellent aggregation brought about by iron oxides while the sticky and plastic consistence can be explained by the high clay content and the kaolinitic clay mineralogy of the soils (Asio, 1996). The excellent soil aggregation also explains the low bulk density and high porosity of the highly weathered soils evaluated.

#### **Soil Chemical Properties**

Highly weathered soils in the humid tropics are generally strongly acidic since they have been subjected to intensive leaching resulting in the excessive loss of bases and nutrients (Sanchez, 1976; Juo and

Franzluebbers, 2003). Highly weathered soils also generally possess variable charge colloids due to the abundance of oxide clays. Mekaru and Uehara (1972) introduced a simple method to determine the net charge of soil colloids and that is the use of pH in H<sub>2</sub>O and pH in KCl. When pH in KCl is lower than pH in H<sub>2</sub>O, it indicates that the soil colloids net charge is negative. Findings of the study revealed that the soils have a net negative charge which implies that the soils can hold and retain positively charged nutrients. The low organic matter contents of the highly weathered soils suggests a low capacity to sequester carbon which can be ascribed to their clay mineralogy that is dominated by iron oxides and kaolinite clays. In terms of P availability, Jahn and Asio (2006) highlighted that P is the most limiting nutrient in tropical soils. This is due to the soils acidic nature, which results in P fixation by iron and aluminum oxides. It could also be due to the low P stock in the soil from intense weathering that occurred. The low exchangeable bases of all soil profiles indicated that the bases have already been lost by prolonged leaching (please see previous discussion). Previous pedological studies in central Philippines (Asio, 1996; Asio and Jahn, 2006; Navarrete et al., 2007) support the findings of the present study.

#### **Soil Classification**

The highly weathered soils studied belong to Ultisols and Oxisols in Soil Taxonomy (Soil Survey Staff, 1999). Ultisols are acidleached soils of warm and humid climates that have a B horizon enriched in clay usually 1:1 and oxide clays. Oxisols are highly weathered soils of warm and humid climates that are infertile and dominated by oxide and low activity clays (Schaetzl and Anderson, 2005). Both soils have low fertility and pose problems for crop production in humid tropical areas especially because they are generally widespread in this part of the world. Soil profiles 1, 2, and 3 have ochric epipedon and oxic endopedon and are classified as Hapludox. Soil profiles 4 and 5 have ochric epipedon and argillic endopedon and are classified as Paleudult. Lastly, soil profiles 6, 7, and 8 have ochric epipedon and argillic endopedon and belong to Hapludult. Paleudults generally have more developed profiles than Hapludults. Soil profiles 4 and 5 are located stable surfaces under rainforests vegetation that have been subject to less human disturbance. This probably explains for their betterdeveloped solum than soil profiles 6, 7, and 8.

## Conclusion

Among the soil profiles, only profiles 3 and 8 have developed from non-uniform or heterogeneous parent materials. Findings also revealed generally heavy losses of K<sub>2</sub>O, CaO, MgO, and Na<sub>2</sub>O from the highly soil profiles evaluated. The amount and profile distribution of K<sub>2</sub>O and CaO is below 0.5% in the entire profile of most soils. On the other hand, there is apparent enrichment of Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, and to a lesser extent SiO<sub>2</sub> in the highly weathered soil profiles thus supporting the residua hypothesis. In terms of morpho-physical characteristics, the soils have deep solum, reddish color, subangular structure, friable moist consistence, and sticky and plastic wet consistence which are all related to the highly weathered nature of the soils. They also generally have low bulk density and higher porosity due to iron oxides aggregation effect. The strong acidity (pH <5) and negative delta pH values revealed that the soil colloids possess a negative net charge. Nutrient status also showed low contents of organic matter, total N, available P, and exchangeable bases. Majority of the deep and highly weathered soils evaluated have possibly developed from homogenous parent materials. The soils are classified as Hapludox, Hapludult, and Paleudult.

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### Integrated use of bio-organic and chemical fertilizer to enhance vield and nutrients content of tomato

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#### Abstract

Excessive use of chemical fertilizers causing a serious threat to the agro-ecological system, developing resistance to pest and declining food safety. Under current scenario, the application of bio-organic nutrients sources become imperative to sustain the productivity of arable farming. Thus to study the possible use of bioorganic sources of nutrients in soil fertility, crop quality and saving the application cost of chemical fertilizer, a pot experiment was conducted in green-house at Land Resources Research Institute, NARC Islamabad. Integrated effects of bio-organic fertilizers such as phosphate solubilizing bacteria (PSB), vermicompost (VC) along with chemical fertilizer was investigated on soil-plant nutrients contents, growth and yield of tomato. Post-harvest results showed that the integrated use of bio-organic fertilizers with chemical fertilizer significantly increased the agronomic yield (Plant height and chlorophyll content) and fruit yield (Number of fruits, fruit weight, fruit diameter and yield) in tomato. The maximum plant height (161.24cm), chlorophyll contents (61.2), number of fruits (19), fruit weight (55g), fruit diameter (45.6a) and fruit yield (1.39 Kg/plant) were recorded in the treatment T5 where VC+PSB+75%RD were applied and minimum in treatment T1 (control). Treatment T5 has increased 117% fruit yield over control. The highest N (2.05% and 2.89%), P (0.33% and 0.50%) and K (2.32% and 6.67%) concentration in shoot and fruit of tomato respectively were found in treatment T5 (VC+PSB+75%RD). Similarly, in soil the highest N (4 mg Kg<sup>-1</sup>), P (0.66mg Kg<sup>-1</sup>) and K (3.53mg Kg<sup>-1</sup>) was recorded in treatment T6 (VC+PSB+100RD). Thus, study results recommend that the integrated use of bio-organic sources with appropriate proportion of chemical/synthetic fertilizers is best option of fertilizer savings and to achieve maximum benefits regarding quality and yield.

Keywords: Tomato, phosphate solubilizing bacteria, vermicompost, chemical fertilizer, greenhouse conditions.

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#### Introduction

Tomato (*Solanum lycopersicum*) is an important crop and is grown throughout the year in different parts of Pakistan. The average yield is 10.3 tons ha-1 (Malik et al., 2018), which is quite low as compared to other countries. While 44-65 tons ha-1 is commercial yield under good management and production practices (FAO, 2020). Bulging population demands increase in the production of tomatoes, which can be met by increasing per unit production. Tomato cultivars are much sensitive to hot climate which is one of the limitations in the optimum production of the summer tomato crop in the plains of Pakistan (Khan et al., 2020). Chemical fertilizers contain salts of ammonium, nitrate, phosphate, potassium and heavy metals in a specific concentration. For better crop production qualitatively and quantitatively, the efficiency of fertilizer use has increased many folds. In Pakistan, formers consider over application of fertilizer only way to

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increase the crop yield. In such a scenario, globally the fertilizer application increased which causing serious environmental issues. Fertilizers not only improve crop production but also increase the accumulation of salts and heavy metals in the soil-plant system. In such conditions, plant also uptake the salts and heavy metals along with the fertilizer absorption from the soil and enter into the food chain. Consequently, fertilization causes soil, water and environmental pollution.

In changing scenario, our interest is towards sustainable agriculture by using biological and organic source for crop production. Biofertilizers increase biological nitrogen fixation and enhance the availability of nutrients to plant (Kaur et al., 2017). Phosphorus plays a key role in plant growth and development. The majority of the soils throughout the world are phosphorus-deficient (Shahid et al., 2012). When chemical fertilizer applies, phosphorus rapidly transformed into less available form by forming a complex with Al or Fe in acid soils with Ca in calcareous soils and become unavailable to plants (Namli et al., 2017). Phosphate solubilizing microorganisms (PSMs) convert insoluble phosphate to soluble forms by acidification, exchange reaction and chelation (Olanrewaju et al., 2017). PSB provide sustained phosphorus supply (Park et al., 2016) along with stimulation of nitrogen fixation and enhance the accessibility of trace elements by synthesizing important growth promoting substances like antibiotics, side spores, etc (Amanullah and Khan, 2015).

Vermicompost (organic manure) supply macro and micronutrients and ultimately improve physical, chemical and biological properties of soil (Chatterjee and Bandyopadhyay, 2014). Vermicompost also helps in nutrients uptake, disease and pest control, and productivity (Barrios-Masias et al., 2011). Several workers reviewed the significant role of vermicompost and PSB, which enhance the yield and at the same time quality of different vegetable crops like tomato (Bihari et al., 2018), chilli (Anggraheni et al., 2019) by influencing the soil properties. So by the application of PSB and vermicompost, we can reduce the hazardous effect of chemical fertilizer by reducing their use, which ultimately turns towards good soil health.

#### **Material and Methods**

This research pertained to study the effect of integrated use of bio-organic and chemical fertilizers on growth, yield and nutrients contents of the tomato plant. A pot experiment was conducted in a greenhouse at National Agricultural Research Center (NARC) Islamabad during fall 2015-2016. NARC is situated at Latitude (33.420 North) and Longitude (73.080 East). The climate of the region varies from semi-arid to subtropical with extreme winter and summer. Vermicompost (VC) and phosphate solubilizing bacteria (PSB), and diammonium phosphate (DAP) were applied as sources of bio-organic and as a chemical fertilizer respectively. Soil samples were collected from the vegetable research farm and vermicompost from Soil Biology Program at NARC, Islamabad. Soil samples were pulverized, air-dried and mixed thoroughly. Before sowing, each experimental pot was filled with 10 Kg soil. Vermicompost was applied @ 05 ton ha<sup>-1</sup> and DAP @ rate of 125 Kg ha<sup>-1</sup>. Before the experimental setting, soil and vermicompost were analyzed for physio-chemical characteristics and given in Table 2 and 3. The two months old tomato seedlings (CV $\approx$  Rio Grande; 15-25cm tall with 3-4 true leaves) were taken from the vegetable program and transplanted in the pots by dipping in PSB solution. The experiment was laid out in a completely randomized design (CRD) with three replications. The treatments detail is given in Table 1.

Table 1. Treatment plan			
Treatments		Descriptions	
T <sub>1</sub>		Control	
T <sub>2</sub>		VC+75% RD	
T <sub>3</sub>		PSB Inoculation+75% RD	
$T_4$		75% RD	
<b>T</b> <sub>5</sub>		VC+PSB+75%Rd	
T <sub>6</sub>		VC+PSB+RD	
Τ <sub>7</sub>		RD	
Table 2. Physiochemical Analy	rsis of Vermicompost.		
Characteristics	Unit	Value	
рН		7.5	
EC (1:1)	dSm <sup>-1</sup>	3.0	
Total N	(%)	2.3	
Total K	(%)	2.0	
Total P	(%)	0.3	
C:N		15.0	
Organic Matter	(%)	25.0	

Soil Characteristics	Unit	Value	
рН		7.80	
EC (1:1)	dSm <sup>-1</sup>	1.70	
Total NO <sub>3</sub> -N	mg/kg	3.20	
Total P	mg/kg	2.33	
Extractable K	mg/kg	101.00	
Organic matter	%	0.81	
Clay	%	20.00	
Sand	%	35.00	
Silt	%	45.00	
Textural Class		Loam	

Table 3. Physio-chemical analysis of soil.

#### Soil and plant chemical analysis

Post-harvest chemical analysis of soil and plant are given in Table 4 and 5. The Hydrometric method was used to determine the soil texture. Soil pH and EC was determined by 1:1 soil-water past using dual pH-EC meter. Soil NO<sub>3</sub>-N, total P and available K was determined by the ammonium Bicarbonate-Diethylene Triamine Penta Acetic Acid (AB-DTPA) method (Soltanpour and Workman, 1979). While concern plant analysis: total nitrogen was measured by Kjeldahl method (Kjeldahl, 1883), P and K were measured by Di-acid (Nitric acid and perchloric acid) digestion method using a spectrophotometer.

#### Results

#### Dynamics of soil and plant chemical analysis

Among different values, treatment comparison showed that soil pH, N, P and K was statistically affected by the different treatments. However, maximum pH 7.67 was recorded in T1 (control) and after that, it was decreased in all treatments and minimum pH (7.27) was recorded in treatment T6 where both organic and inorganic fertilizer applied (Table 4). In the case of soil P content, the highest P was observed in combining the application of T6 (VC +PSB+RD) followed by T5 (VC +PSB +75% of RD) and minimum in treatment T1. Overall soil P contents were increased with PSB, VC and fertilizer application. Similarly, soil N and K content were also improved in all treatments. Maximum N and K Contents was recorded in T6 and minimum in treatment T1 (Control). The decrease in pH is due to the release of organic acids by the reactions of VC and PSB. Likewise, the nutrient concentration of NPK also improved in soil due to the production of organic acids which results in more release of nutrient from the soil. The results are in line with the findings of Kumar et al. (2001), who reported that biofertilizers have the ability to increase nutrient bioavailability through a biological process which ultimately increases yield. Many studies clearly concluded that the application of biofertilizer and vermicompost improved soil health and nutrient availability (Maji, 2013; Rani and Jha, 2020).

Table 4. Chemical analysis of soil on experiment harvest.	
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Treatments	рН	Total phosphorus (mg kg <sup>-1</sup> )	Total nitrogen (mg kg-1)	Extractable potassium (mg kg <sup>-1</sup> )
T1	7.67 a	0.053 c	3.20 e	2.40 a
T2	7.63 a	0.074 c	3.80 bc	3.00 bc
Т3	7.57 ab	0.125 bc	3.60 cd	3.17 ab
T4	7.37 cde	0.058 c	3.40 d	2.71 ab
T5	7.43 cd	0.134 b	3.90 b	3.40 abc
Т6	7.27 efg	0.066 b	4.20 a	3.53 c
Τ7	7.47 bc	0.059 c	3.90 b	2.80 b

#### Effect of vermicompost, PSB and chemical fertilizers on nutrient contents of tomato

In tomato, whole shoot and fruit analysis of nitrogen, phosphorus and potassium showed that their concentration was significantly affected by treatments given in Table 5. In the case of tomato shoot analysis, the mean highest nitrogen (2.05%), phosphorus (0.33%) and potassium (2.32%) were recorded in T5 (VC+PSB+75% RD), followed by T6 (VC+PSB+RD). Meanwhile, the mean lowest value of N (1.45%), P (0.15%) and K (1.81%) was noted in T1 (control). It was recorded that all the treatment significantly influence the NPK contents of the tomato shoot but the influence of treatment T5 was recorded clearly at par as compared to all other treatments. Similarly, in the case of tomato fruit analysis, the mean maximum nitrogen (2.89%) phosphorus (0.50%) and potassium (6.67%) was recorded in treatment T5 (VC+PSB+75% RD), followed by treatment T6 (VC+PSB+RD) and mean the lowest value of N, P and K were recorded in T1 (control).

Treatments	1	Tomato whole shoot analysis			Tomato Fruit analysis		
	N, %	P, %	К, %	N, %	P, %	К, %	
T1	1.45 i	0.15 g	1.83 h	2.12 h	0.25 g	4.01 c	
T2	1.94 fg	0.31 bc	2.20 f	2.65 d	0.46 c	4.83 bc	
Т3	1.86 g	0.33 de	1.91 g	2.48 e	0.36 e	5.91 a	
T4	1.99 ef	0.20 f	2.24 ef	2.27 g	0.30 f	5.01 b	
Т5	2.05 cde	0.33 b	2.32 d	2.89 c	0.50 b	6.67 a	
Т6	2.03 def	0.30 bc	2.27 de	2.66 d	0.41 d	6.01 a	
Τ7	1.69 h	0.18 fg	2.29 de	2.38 f	0.30 f	4.65 bc	

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#### Effect of vermicompost, PSB and chemical fertilizers on agronomic parameters of tomato

The influence of different treatment composition on agronomic parameters of tomato is shown in Table 6. The maximum mean plant height was 161.24 cm was recorded in T5 (VC+PSB+75% RD), followed by 154.16 cm in T6 (VC+PSB+RD). The mean lowest plant height was 102.23 cm was recorded in T1 (control), followed by 115.10 cm in T7 (RD). The mean maximum chlorophyll content was 61.2 was noted in T5 (VC+PSB+75% RD), followed by 59.0 in T6 (VC+PSB+RD). On the other hand mean lowest chlorophyll content was 39.7 in T1 (control), followed by 55.2 in T2 (75% RD). Similarly, the mean maximum number of fruits was 19 in T5 (VC+PSB+75% RD), followed by 18 in T6 (VC+PSB+RD). Mean lowest fruits were recorded 10 in T1 (control), followed by 13 in T2 (VC+75% RD). The fruit diameter was measured in milli meters (mm). The mean highest diameter of fruit was 45.6 mm in T5 (VC+PSB+75% RD), followed by 42.2 mm in T6 (VC+PSB+RD). While the lowest recorded diameter was 29.4 mm in T1 (control), followed by 35.1 mm in T3 (PSB Inoculation+75% RD). Fruit weight was measured in g kg<sup>-1</sup>. The mean maximum fruit weight was 55.0 g kg<sup>-1</sup> was recorded in T5 (VC+PSB+75% RD), followed by 52.9 g kg<sup>-1</sup> in T6 (VC+PSB+RD). On the other hand, the minimum mean value of 17.5 g kg<sup>-1</sup> was recorded in T1 (Control). In the case of fruit yield, the mean maximum yield was 1.39 ton ha-1 recorded in T5 (VC+PSB+75% RD), followed by 1.32 ton ha-1 in T6 (VC+PSB+RD). On the other hand mean minimum value was 0.64 ton ha<sup>-1</sup> in T1 (control), followed by 0.96 ton ha<sup>-1</sup> in T2 (VC+75% RD) shown in Table 6.

Trea	atments	Plant Height	Chlorophyll	No. of fruits	Fruit weight	Fruit diameter	Yield
		(cm)	content		(g)	(mm)	(Kg)
T1	Control	102.23 j	39.7 i	10 f	17.5 i	29.4 h	0.64 h
T2	75% RD	124.16 h	55.2 f	13 de	39.0 f	39.2 c	0.96 e
Т3	PSB+75%RD	140.85 de	56.4 de	16 b	49.6 cd	35.1 efg	1.13 d
T4	VC+75% RD	144.16 cd	56.5 cd	15 bc	48.3 de	36.2 ef	1.14 d
T5	VC+PSB+75%RD	161.24 a	61.2 a	19 a	55.0 a	45.6 a	1.39 a
T6	VC+PSB+RD	154.16 b	59.0 b	18 a	52.9 ab	42.2 b	1.32 b
T7	RD	115.10 i	56.5 d	14 cd	39.3 f	38.7 cd	1.26 c

Table 6. Effect of treatments on	agronomic parameter of tomato
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#### Discussion

It has been recorded that not only tomato growth and yield but also the soil properties (physical and chemical) were significantly affected by the addition of organic and inorganic sources to the soil. Tomato has high production ability 44-65 tons ha<sup>-1</sup> under good management and production practices (FAO, 2020). But due to imbalance in soil nutrients and extreme environmental changes, production cannot be achieved at the expected rate. However, with the addition of external nutrients sources to soil through organic and inorganic amendment and using integrated nutrient management practices, soil health can be improved (Jack and Thies, 2006). Micronutrients also supplied to soil by organic source which cannot be supplied by chemical fertilizers (Umar et al., 2008). The use of biofertilizers as an alternate of chemical input can be ensured the bioavailability of nutrients through biological processes, and increase vegetable yield (Kumar et al., 2014) and fruits production (Maji and Das, 2008). Proper use of organic and inorganic amendment will help to maintain sustainability in quality and production by maintaining soil fertility by reducing health hazards (Pal et al., 2015; Palaniappan and Annadurai, 2018), at the same time help to control insect-pest diseases (El-Gleel Mosa et al., 2014; Maji, 2013). Evidences showed that the PGPR application increases the availability of nutrients in the rhizosphere for the plant uptake (Kumar et al., 2015; Etesami and Adl, 2020).

Tognetti et al. (2005) reported that vermicompost highly tended to lower the pH of soil than simple compost. Maximum pH was recorded in control and reduced in all the other treatment of the experiment because in the presence of the biofertilizer and activities of PSB, acids were produced in close area that reduce the pH of the medium. Similar results were stated by El-Tarabily et al. (2006) that application of

oxidizing bacteria enriched biofertilizer reduced the pH of the soil as compared to control. Similar results were recorded by the Berger et al. (2013) and Lima et al. (2010), application of biofertilizer with microorganism comparatively reduced the pH of the soil over control. In soil, phosphorus is present in an insoluble form, which is not available to plants (Tognett et al., 2007). Same time, in the rhizosphere many organism's present i.e. PSB (Nautiyal et al., 2000) which secretes acids (amino) for the bioavailability of phosphorus (Kim et al., 1998; Richardson, 2001). Organic acids and hormones increase the soil phosphorus availability and ultimately plant growth increase (Chatterjee and Bandyopadhyay, 2014). Fertilizers, organic manures and PSB when applied in combination were shown best result and increase phosphorus availability (Ughade et al., 2016). Vermicompost contains a significant amount of nitrogen and which is easily mineralized and available for uptake of plants (Barani and Anburani, 2004). Application beneficial microorganisms enhanced the bioavailability of nutrients in brinjal (Prabhu et al., 2004), in cucumber (Mali et al., 2005). Vermicompost contains nitrogen in nitrate form instead of ammonia (Chatterjee and Bandyopadhyay, 2014). In current study, combined application of chemical fertilizers, organic manures and PSB increased nitrogen availability (Ughade et al., 2016). In vermicompost considerable amount of potassium is present which easily available to plants. In results, bio-fertilizers application also increased the availability of potassium to plants. A field study stated that application of PGPR and inorganic fertilizer increased the uptake of K in plant by 58% while N and P three times as compared to control in wheat (Abbasi et al., 2011). Khalig et al. (2006) stated that a significant increase in the uptake of K (10-15%) recorded in cotton plant on the application of PSB containing PGPR amended with bioorganic and chemical fertilizers over control. Application of vermicompost and PSB together greatly improved the efficiency nutrients availability (Chatterjee and Bandyopadhyay, 2014). Result showed that plant height and number of leaves increased significantly because in the rhizosphere bacteria produced plant growth regulators which increased chlorophyll contents. So increase in plant height, the number of leaves and chlorophyll content was due to the nitrogen fixation of bacteria (Douds et al., 2006). An increase in plant height, number of leaves, number of flowers, stem girth and chlorophyll content were reported by use of vermicompost and PSB (Nazir et al., 2006; Tripathi et al., 2014; Damse et al. 2014). It has been reported that combined application of vermicompost and PSB along with lower fertilizer dose increased the numbers of flowers, chlorophyll content, numbers of leaves and fruits (diameter, weight and number (Singh et al., 2015b; Soni et al., 2018). Fertilizers, organic manures and PSB when applied in combination were shown the best result and plant yield and growth in tomato (Singh et al., 2015a). The same results were obtained in chilli by Khan et al. (2012). Yeptho et al. (2012) recorded that 50% NPK + PM + biofertilizer application increases the leaf area, the number of leaves, fruit weight and yield.. By the combined application of vermicompost and PSB increase plant height, flowering and fruit yield in chilli (Vimera et al., 2012); in radish (Deepika et al., 2010) and in strawberry (Singh et al., 2008).

#### Conclusion

The results of the present study concluded that the application of PSB enriched bio-organic (vermicompost) fertilizer could help to reduce the overuse of costly synthetic fertilizers. Application of PSB and vermicompost significantly reduce the cost of production without affecting the yield. Moreover, the use of PSB and vermicompost with a lower rate of chemical fertilizer lead to improvement of tomato growth and yield traits as well as soil and plants nutrients status. Therefore, it is concluded that the application of bio-organic fertilizers as a supplement can reduce the rate as well as the cost of synthetic fertilisers. Thus, through the integrated use of organic and inorganic sources of nutrients, the expected yield of tomatoes can be achieved keeping in view the healthy soil and environmental aspects.

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# Soil properties and grape yield of vineyard as affected by sawdust addition in a semi-arid region

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#### Abstract

Due to its high organic matter content, sawdust can be used for soil amendment. This study investigated the effect of sawdust obtained from a furniture industry (Yildiz Integre Akhisar flakeboard) on grape yield and loam texture soil (Typic Xerofluvent) properties of a vinevard located in Saruhanlı District, Manisa, Turkey. In the trial, three groups with dry sawdust (10 t ha-1, 20 t ha-1; 30 t ha-1) was added to plots of vinevard while 0 t ha<sup>-1</sup> or without sawdust addition was set up as a control group. All the 4 treatments were replicated three times (4×3). Sawdust addition resulted in significant increase (p <0.05) of soil organic matter, total nitrogen, electrical conductivity (EC), available Na, Ca, Mg, Fe, Zn, P, K, and Mn contents, moisture, saturation percentage, field capacity, wilting point, available water and aggregation percentage values compared with control group. Also, the soil pH, bulk density, and dispersion percentage significantly decreased with sawdust addition. However, there was no significant change in lime, available Cu contents, structure stability index, aggregate stability of the soil with sawdust addition. Compared with control group, addition of 20 t ha-1 and 30 t ha-1 of dry sawdust significantly increased the grape yield of the vineyard. The highest yield of 4.23 t ha-1 was recorded with 30 t ha-1 sawdust addition. Sawdust can be used as a soil amendment given its positive effects on some soil properties and grape yield of the vineyard. It is suggested that nitrogen immobilization from the soil by microorganisms due to sawdust's high C/N ratio is a key consideration for its future use.

Keywords: Grape yield, loam texture soil, sawdust, semi-arid region.

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#### Introduction

The benefits of soil organic matter (SOM) have been largely explored by many studies (Krull et al., 2004). Organic matter (OM) is known to improve soil structure, supply macronutrients, and micronutrients, and limit soil erosion. Also, it has the potential to increase soil water holding capacity that is a prominent aspect, particularly, in semi-arid regions (Medrano et al., 2015). The effect of organic matter on soil properties is related to its amount (Murphy, 2014).

The organic matter content in Turkey's soils, especially Egean region is generally low (Eyüpoğlu, 1999). Many studies carried out in the Egean region in Manisa districts (Alaşehir, Salihli, Akhisar-Gölmarmara) revealed that organic matter content of vineyard soils is less than 1% (Merken and Önder, 2014; Çoban et al., 2016; Yağmur and Okur, 2018). Thus, it appears relevant to increase its amount in the soil to obtain the expected benefits. Increasing the amount of SOM, and thereby the water holding capacity could lead to a reduction of irrigation water use that is generally limited in semi-arid regions (Medrano et al., 2015). Moreover, Yagmur et al. (2014) showed that irrigation water for vineyards in Alasehir, a district of Manisa, could present salinity and boron problems. Therefore, the use of organic materials to optimize water use is of great importance.



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Several organic materials (e.g., sawdust, rice bran, cocoa husk ) are used to increase SOM (Olayinka and Adebayo, 1985; Moyin-Jesu, 2007). Sawdust consists of the dust and small pieces of wood resulting from woodworking operations and it has a very high carbon to nitrogen ratio (more than 100) (Allison, 1965). Large loads of wood chip are produced in the world. For instance, nearly 58 million metric tons of wood waste were produced in the United States in 2011 making a cumulative amount of about 3.5 billion metric tons (EPA, 2018). There is wood industry in almost every province of Turkey (Yildirim, 2012). The resulting sawdust is generally sent to pellet fuel plants, chipboard, and flooring product factories. Utilization of unused sawdust in agricultural sector can be beneficial to the soil and plant thus, contribute to a circular economy by closing the nutrients loop (Hannah, 2007).

The use of sawdust in agriculture goes back to several years ago. It can be used as animal bedding or be applied to the field as mulch or soil improver (Allison and Anderson, 1951). The sawdust can be applied to field in different forms: as compost after being processed with other materials (Aggelides and Londra, 2000), as ash (Awodun, 2007; Moyin-Jesu, 2007), or simply in its raw form, very often, supplemented with inorganic fertilizers or manure (Turk, 1943; Olayinka and Adebayo, 1985; Tran, 2005; Gali, 2011). This supplementation takes into account the high C/N ratio of sawdust that may lead to N immobilization from soil by microorganisms for the mineralization purpose (Sawada et al., 2015). The C/N ratio and the amount of other elements present in sawdust depend on the wood from which it is derived (Viljoen and Fred, 1924; Allison, 1965). Many studies emphasized the increase of crop yield and improvement of soil physical and chemical properties due to addition of sawdust and its derived products either alone or in combination (Olayinka and Adebayo, 1985; Aggelides and Londra, 2000; Chiroma et al., 2006; Awodun, 2007). These effects are more pronounced over a long period.

In most studies, the crops assessed are generally annual (e.g., okra, barley, beans, etc.). Few studies have been carried out to explore the benefits of sawdust addition to perennial crops such as vineyards, less in a semi-arid context. According to FAOSTAT (2019), Turkey ranked 5th and 6th worldwide in vineyards area and production of grapes, respectively. Almost half of the country's vineyards are located in the semi-arid Aegean region, particularly in Manisa province. Therefore, the current study investigated the effect of fine sawdust addition on soil properties and grape yield of a vineyard in Saruhanlı district of Manisa province.

#### **Material and Methods**

#### Experimental field and sawdust

The experiment was carried out in a vineyard of about 2000 m<sup>2</sup> belonging to a local farmer in Saruhanlı district of Manisa province. Saruhanlı district has a warm and temperate climate. There is more rainfall in the winter than in the summer. According to the Köppen-Geiger climate classification, it can be called Csa (Mediterranean climate with warm winter, very hot and dry summers). The average annual temperature and rainfall in the region is 16.6 ° C and 665 mm, respectively (Climate, 2019). The soil was loam texture slightly alkaline. Pre-trial soil analysis of some properties in the study field are shown in Table 1.

Parameters		Units	Value
рН	-		7.52
Electrical conductivity		µmhos cm <sup>-1</sup>	875.33
Lime		%	9.50
Organic matter (OM)		%	1.62
Sand		%	37.12
Clay		%	36.72
Silt		%	26.16
Total N		%	0.11
	Р	mg kg <sup>-1</sup>	23.92
Available	К	mg kg <sup>-1</sup>	461.94
	Са	mg kg <sup>-1</sup>	5583.33
	Mg	mg kg <sup>-1</sup>	541.00
	Mn	mg kg <sup>-1</sup>	18.10
	Fe	mg kg <sup>-1</sup>	4.28
	Cu	mg kg <sup>-1</sup>	4.73
	Zn	mg kg <sup>-1</sup>	1.06
	Na	mg kg <sup>-1</sup>	58.88

Table 1. Soil characteristics at the start of the experiment

Table 2. Characteristics of the sawdust

The sawdust used in the trial was obtained from a flakeboard fabric industry (Yıldız Entegre Ağaç Sanayi ve Ticaret AŞ) located in Manisa-Akhisar. The sawdust had a fine structure since it was a waste of flakeboard processing. The sawdust used in the trial and its characteristics are shown in Figure 1 and Table 2, respectively.



Figure 1. Sawdust used in the experiment

Parameters		Units	Value	
рН		-	7.76	
Electrical conductivity		µmhos cm-1	1611.00	
Lime		%	3.73	
Organic matter		%	67,59	
C/N		-	71,28	
	Р	mg kg <sup>-1</sup>	6.00	
	Na	mg kg <sup>-1</sup>	806.00	
	К	mg kg-1	1129.00	
Available	Ca	mg kg-1	8820.00	
Available	Mg	mg kg <sup>-1</sup>	712.00	
	Fe	mg kg <sup>-1</sup>	51.76	
	Cu	mg kg-1	7.05	
	Zn	mg kg <sup>-1</sup>	50.82	
	Mn	mg kg <sup>-1</sup>	13.22	
	Ν	%	0.55	
	В	mg kg-1	28.30	
	Р	mg kg <sup>-1</sup>	655.00	
	Na	mg kg-1	1032.00	
	K	mg kg-1	4365.00	
Total	Са	mg kg <sup>-1</sup>	22295.00	
Total	Mg	mg kg-1	4530.00	
	Fe	mg kg-1	7035.00	
	Cu	mg kg <sup>-1</sup>	7.46	
	Zn	mg kg <sup>-1</sup>	147.00	
	Mn	mg kg <sup>-1</sup>	174.00	

#### **Experiment establishment**

The sawdust was collected from the plant in Manisa Akhisar in April 2018. It was manually applied all around the plants in Manisa Saruhanlı on the same date. The treatments consisted of control without any application (0 t ha<sup>-1</sup>, T), 10 t ha<sup>-1</sup> (1), 20 t ha<sup>-1</sup> (2), 30 t ha<sup>-1</sup> (3) of dry fine sawdust. Applications were made after the vineyard was pruned (in February 2018) and the field was ploughed. After the sawdust was applied to the soil surface, it was covered with soil by the mean of a tractor. The trial was set up following a randomized complete block design (RCBD) with three replications as shown in Figure 2. Each plot was constituted of three grape trees. The variety of grape used was the seedless sultana. The sultana grape is known to be juicy, sweet and refreshing.



T: Control No application, 1: 10 t ha<sup>-1</sup>, 2: 20 t ha<sup>-1</sup>, 3: 30 t ha<sup>-1</sup> of dry sawdust Figure 2. View of the trial plan.

#### Soil analysis

Soil sampling was done at the start and approximately 6 months after addition of sawdust (September 2018) and collected at a depth of 0-15 cm. Pre- and post-trial soil analysis of available Fe, Zn, Cu, and Mn were determined by diethylenetriaminepentaacetic acid (DTPA) extraction method (Lindsay and Norvell, 1978). Determination of available Na, Ca, Mg, K was carried out by ammonium acetate (1 N NH<sub>4</sub> Oac pH:7) method (Kacar, 1995). Available P was analyzed using Olsen method (Olsen et al.,1954). Organic Carbon (OC) was determined by the procedure of Walkley and Black using the dichromate wet oxidation method (Nelson and Sommers, 1996). OM was calculated by multiplying OC content by 1.724. The modified Kjeldahl method was used for determination of total nitrogen (Bremner, 1965). Bulk density was estimated by cylinder method (Black, 1965) and pore space saturation method (Richards, 1954), respectively. A pressure plate was used to determine field capacity and wilting point at pF of 2.54 and 4.2, respectively. Available water was calculated by the difference between these two features (Richards, 1954). A pH meter was used for soil pH measurement (Jackson, 1967). The calcimetric method was applied for lime measurement. (Soil Survey Staff, 1951). Aggregation percentage, dispersion percentage, structure stability index, and aggregate stability were calculated using methods presented by Kemper and Roseneau (1986).

#### Determination of grape yield

The grape harvest was carried out in September 2018. The grape yield from the different plots was determined by weighing.

#### Statistical analysis

The collected data were subjected to variance analysis (ANOVA) using the R 3.5.1 version software. Comparison of means was performed with Duncan's multiple comparison test at a significance level of  $\alpha$  = 0.05.

#### **Results and Discussion**

#### Effect of sawdust addition on soil chemical properties

In the study, sawdust application significantly affected soil chemical properties (p <0.05) (Table 3). Table 3. Soil chemical properties at the end of the study

			) + h	a-1		1	0 t	ha-1			20 1	tha-1		2	0+	ha-1		
		Ľ	ιu	d -		1	υι	lla -			20	l IIa -		J	υι	lla -		
pН		7.60	±	0.03	А	7.40	±	0.05	AB	7.30	±	0.04	В	6.80	±	0.10	С	***
EC,µ	umhos/ci	n <b>878.30</b>	±	25.6	D	941.70	±	22.5	С	981.70	±	10.4	В	1033.30	±	25.60	В	***
OM,	%	1.70	±	0.08	D	2.30	±	0.15	С	2.80	±	0.1	В	3.50	±	0.30	В	***
Lim	e, %	9.75	±	0.39	А	9.83	±	0.60	А	9.52	±	0.09	А	9.49	±	0.02	А	ns
Tota	al N, %	0.101	±	0.01	С	0.113	±	0.01	В	0.117	±	0.01	В	0.131	±	0.01	В	***
	Р	23.92	±	5.13	В	29.18	±	0.93	AB	31.59	±	1.20	А	32.28	±	1.59	А	*
7	К	445.79	±	19.38	В	465.17	±	16.79	AB	471.63	±	5.60	AB	484.70	±	9.69	AB	*
,kg	Са	5583.3	±	49.41	В	6059.60	±	58.24	А	6092.5	±	15.82	Α	6130.10	±	20.02	А	***
gm	Mg	514.2	±	9.77	С	527.70	±	6.03	BC	539.0	±	2.89	AB	545.00	±	4.69	AB	**
le,	Na	57.19	±	5.83	С	63.92	±	2.91	BC	68.97	±	2.92	В	77.38	±	5.83	В	**
lab	Fe	4.28	±	0.26	С	5.10	±	0.19	В	5.32	±	0.26	В	6.49	±	0.08	В	***
vai	Zn	1.06	±	0.14	В	1.20	±	0.02	А	1.24	±	0.02	А	1.31	±	0.03	А	*
Ą	Cu	4.73	±	0.50	А	4.99	±	0.24	А	4.93	±	0.66	А	5.05	±	0.15	А	ns
	Mn	20.77	±	0.31	С	22.69	±	7.58	С	34.03	±	4.40	В	45.24	±	1.44	В	***

\*, \*\*, \*\*\* indicate significance at  $p \le 0.05$ , 0.01, and 0.001, respectively. ns is meant for « not significant » The means (n=3) ± standard deviations in each column followed by a different letter are significantly different according to Duncan's test at \*  $p \le 0.05$ .

The soil organic matter (SOM) increased in parallel with increasing sawdust rates. The organic matter content ranged between 1.67% and 3.50% resulting in an increase of 110%. The highest and lowest values were obtained from plots with the highest rate (30 t ha<sup>-1</sup>) and plots without any sawdust addition, respectively. Similar results were obtained in many other studies when organic materials were applied (Demir and Gülser, 2015; Delibacak and Ongun, 2016; Alvarenga et al., 2017). This result could be attributed to the high organic matter content of sawdust (approximately 68%) used in the experiment.

The impact of sawdust addition on soil electrical conductivity (EC) was similar to SOM. Soil EC increased with sawdust increasing rates reaching a value of 1033  $\mu$ mhos cm<sup>-1</sup> in plots that received the highest rate (30 t ha<sup>-1</sup>). This could have resulted from the high electrical conductivity of the added sawdust (Table 2; 1611  $\mu$ mhos cm<sup>-1</sup>). Another reason could be attributed to the release of nutrients as a result of sawdust mineralization that affected the EC (Smith and Doran, 1997).

In comparison with the control, soil nutrients such as total nitrogen (N), available phosphorus (P), potassium (K), calcium (Ca), magnesium (Mg), sodium (Na) iron (Fe) zinc (Zn) manganese (Mn) increased with sawdust addition. It appeared that the highest rate lead to more pronounced impact on soil nutrients. The increments due to addition of 30 t ha<sup>-1</sup> compared with control reached 0.03%, 35%, 8.72%, 9.8%, 3.72%, 35.30%, 34%, 23.6%, 117% for total nitrogen, available P, K, Ca, Mg, Na, Fe, Zn respectively. For soil available P, K, Ca, Zn content, no significant statistical difference was found between the treatments 10 t ha<sup>-1</sup>, 20 t ha<sup>-1</sup> (p <0.05). The increase of these nutrients in the soil can be explained by their presence in the sawdust as indicated in Table 2. In addition, mineralization process probably contributed to the increase of the available nutrients in the soil. However, it appears difficult to conclude since mineral nitrogen was not analyzed in this study.

Soil pH decreased from 7.6 in the control plots to 6.8 in plots with 3 t ha<sup>-1</sup> sawdust addition. This is possible due to several organic acids and H<sup>+</sup> ions produced by organic matter decomposition. Moreover, the carbon dioxide (CO<sub>2</sub>) that results from the decomposition could react with the water present in the soil to form a carbonic acid (H<sub>2</sub>CO<sub>3</sub>) that increases the acidity (Sağlam, 1997). Several studies have reported the decrease of the soil pH as a result of organic materials application to the soil (Candemir and Gülser, 2010; Tavali et al., 2013). No statistical difference was found out between treatments regarding lime and Cu content.

#### Effect of sawdust addition on soil physical properties

Changes in soil physical characteristics as affected by sawdust application are shown in Table 4. Table 4. Soil physical properties at the end of the study

	<b>0</b> t	t h	a <sup>.1</sup>		1	0 t	ha <sup>.1</sup>			20 1	t ha <sup>.1</sup>		3	80 t	ha <sup>.1</sup>		
BD,g cm <sup>-3</sup>	1.33	±	0.03	А	1.19	±	0.04	В	1.14	±	0.02	В	1.06	±	0.03	С	***
M,%	9.27	±	0.61	С	11.37	±	0.69	В	14.13	±	1.11	А	15.22	±	0.24	А	***
SP, %	45.60	±	1.54	С	48.80	±	0.36	В	50.10	±	0.30	В	52.30	±	0.95	А	**
FC, %	30.79	±	0.33	D	32.43	±	0.24	С	33.83	±	0.67	В	35.36	±	0.29	А	***
WP, %	16.20	±	0.79	В	17.20	±	0.40	Α	17.70	±	0.17	А	18.10	±	0.28	А	*
AW, %	14.54	±	0.73	С	15.16	±	0.30	BC	16.10	±	0.59	AB	17.26	±	0.52	А	**
SSI, %	36.67	±	1.15	А	38.00	±	0.00	Α	36.67	±	1.15	А	36.67	±	2.31	А	ns
AS, %	10.88	±	1.97	А	10.61	±	1.93	А	10.74	±	1.09	А	10.51	±	0.92	Α	ns
DP, %	36.57	±	1.73	А	32.72	±	0.00	AB	33.52	±	0.69	AB	31.90	±	2.87	В	**
AP, %	63.43	±	1.73	В	67.28	±	0.00	AB	66.48	±	0.69	AB	68.10	±	2.87	Α	*

BD: Bulk density, M: Moisture, SP: Saturation percentage, FC: Field capacity, WP: Wilting point, AW: Available water, SSI: Structural stability index, AS: Aggregate stability, DP: Dispersion percentage, AP: Aggregation percentage

\*, \*\*, \*\*\* indicate significance at  $p \le 0.05$ , 0.01, and 0.001, respectively. ns is meant for « not significant » The means (n=3) ± standard deviations in each column followed by a different letter are significantly different according to Duncan's test at \*  $p \le 0.05$ .

Similar to soil chemical characteristics, sawdust addition led to statistically significant changes in soil physical properties. Both soil bulk density and dispersion percentage decreased following the sawdust addition. The bulk density was 1.33 and 1.06 (g cm<sup>-3</sup>) in the control and 30 t ha<sup>-1</sup> added plots, respectively representing a decrease of nearly 20%. The lower density of the fine sawdust incorporated into the soil could explain the soil bulk density decrease (Mylavarapu and Zinati, 2009). Furthermore, sawdust addition is associated with increase of soil aggregation and porosity (Eibisch et al., 2015).

Regarding the dispersion percentage, the decrease recorded between the control and the treatments with 30 t ha<sup>-1</sup> was approximately 13%. The decrease in the dispersion rate following application of organic materials has been reported by Turgut and Aksakal (2010). In our study, although the decrease was due to sawdust application, the dispersion rate in all plots varied between 31.90% and 36.57%, indicating that all of the soils remained sensitive to erosion. Soils with a dispersion percentage greater than 15 are likely to be eroded (Bryan, 1968).

Conversely, all characteristics related to soil water such as moisture, saturation percentage, field capacity, wilting point, and available water increased by 64%, 14%, 15%, 12%, 19%, respectively in the plots that received 30 t ha<sup>-1</sup> of sawdust compared with control (0 t ha<sup>-1</sup>). This increase may be attributed to the increase in total porosity of the soil with sawdust addition and thus more water retention of the soil (Khaleel et al., 1981). In addition, sawdust can hold moisture because of its hydrophilic properties (Stevenson, 1982). The soil water content is positively correlated to the amount of organic matter (Barzegar et al., 2002; Khan et al., 2006). Available water is the water held in soil between its field capacity and permanent wilting point (Romano and Santini, 2002). Also, as the increase of field capacity with sawdust is higher than the increase in the wilting point, there is an increase in the available water values.

The effect of sawdust addition on aggregation percentage was found to be statistically significant (P <0.05). The percentage of aggregation of the soil was 63.43% and 68.10% in control and 30 t ha<sup>-1</sup> sawdust added plots, respectively. The effect on the structure stability index and the aggregate stability of the soil was not statistically significant (P <0.05). However, it has been reported that organic matter increases the structural stability index of the soil and aggregate stability (Cercioglu et al., 2014). Since organic matter acts as a cementing agent in the soil, it promotes the flocculation of soil particles and stable aggregate. But given that our trial period was not long enough, sawdust could not significantly affect the structural stability index and the aggregate stability of the soil.

#### Effects of sawdust addition on grape yield

The grape yield was affected by sawdust addition as shown in Figure 3.



Sawdust rates



Error bars are meant for standard deviations. Bars with similar letter(s) are not significantly different at  $P \le 0.05$  using Duncan's test. Figure 3. Yield variation according to the sawdust rates

The grape yield increased with an increasing sawdust rate. However, no statistical significance was found between the control and 10 t ha<sup>-1</sup> treatments. Yields of 3.96 t ha<sup>-1</sup> and 4.23 t ha<sup>-1</sup> were recorded with 20 t ha<sup>-1</sup> and 30 t ha<sup>-1</sup> treatments representing an increase of nearly 12 and 17%, respectively compared with control. Johnson (1944) reported tomato yield increase with sawdust addition. Also, Gali (2011) observed an increase of shoot dry matter of maize due to 5 t ha<sup>-1</sup> and 10 t ha<sup>-1</sup> sawdust application on sandy soil in the short term. The improvement of physical and chemical soil properties driven by the applied sawdust positively affected plant development and grape yield. The addition of sawdust more likely supplied the available nutrients resulting in a yield increase as compared with unamended plot. It appeared difficult to figure out the soil characteristics that impacted the most the yield since neither the subsurface soil where the roots of trees can reach nor the plant itself could be analyzed in this study. However, the increase of soil water content driven by sawdust could be an important aspect, particularly in this semi-arid study area where around 5 mm of rainfall is recorded in two fructification months, July and August. Data could not allow assessing the mineralization dynamic on the study period (about 6 months). However, the addition of nitrogen may be necessary in the future to promote its decomposition in the soil by microorganisms.

#### Conclusion

The effect of fine sawdust waste on loamy soil properties and grape yield was explored in this study. Sawdust addition led to improvement of soil properties and grape yield. In terms of the chemical properties, sawdust significantly increased the SOM, total nitrogen, EC, available Na, Ca, Mg, Fe, Zn, P, K, and Mn content. Conversely, a decrease in soil pH was recorded with sawdust addition. The lime and available Cu content of the soil did not show a statistically significant change. Regarding the soil physical properties, sawdust addition statistically increased the soil moisture, saturation percentage, field capacity, wilting point, aggregation percentage, and available water. An inverse effect was observed on the soil bulk density and dispersion percentage. Soil stability index aggregate stability was not statistically significant. Grape yield increased with sawdust addition compared with control group (without sawdust addition). The highest grape yield was determined in 30 t ha<sup>-1</sup> sawdust addition. There was no statistical difference between the control and 10 t ha<sup>-1</sup> treatment for grape yield. The use of sawdust as a soil amendment appears an interesting management. Nevertheless, its high C/N ratio could lead to negative effects if nitrogen is not supplemented in the soil.

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# Effect of different organic wastes on biological properties of maize (Zea Mays Indendata) rhizosphere

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#### Abstract

This study was carried in order to determine the effects different various organic wastes (tobacco prodction waste, wheat straw, tea waste and hazelnut husk) under greenhause conditions on biological properties (microbial biomass C, basal soil respiration, dehydrogenase activity, urease activity and arlysulphatase activity) in clay-loam soil and rhizosphere (Zea mays indandata) soil of maize plant. The organic wastes were thoroughly mixed with the soil at a rate equivalent to 50 g kg<sup>-1</sup> on airdried weight basis. Experimental desing was randomized plot desing with there replications in greenhause. The moisture content in soil was mantained around 60 % of maximum water holding capacity by weighing the pots everday. Changes in the biological properties were determined in the soil and rhizosphere (Zea mays indendata) samples and root free soil taken in 15, 30, 45, 60, 75 and 90 days after the experiment was conducted. At the end of experiment, all organic waste added soil increased biological properties of soil in comparison with the control (P<0.01) at all experimental periods. Moreover, biological properties in rhizosphere soil were higher than in root free soil at all organic waste application (P<0.01). Increased of organic wastes on soil biological properties had different trend (P<0.01), the most increases in the biological properties in the soil treated with tea wastes and tobacco production waste with supplying of low initial C/N ratio compared to other organic wastes.

**Keywords**: Organic waste, soil, rhizosphere, microbial biomass, basal soil respiration, enzyme activities.

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#### Introduction

The loss of soil organic matter under intensive land use is one of the many factors that degree agricultural soil of Anatolia. Traditional agricultural practices also leads to decrease fertility and, therefore, to declining productivity (Kenenbayev and Kucherov, 1994; Kızılkaya 2004). Soil organic matter is extremly heterogenous ranging from only slightly decomposed plant and microbial residues to higly humified organic substances. The most common practice to preserve and/or restore soil fertility is to add organic matter, which, preferentially, should be sufficiently stabilized to produce beneficial effects (Gallardo-Lara and Nogales, 1987; Mathur et al., 1993). Therefore, different types of organic wastes have increasingly been applied to soils in recent years. Organic wastes applications haven't only increased the soil organic matter, but have also enhanced the soil's C and N contents, and have improved biological activity in soil (Vigil et al., 1991). Plants influence C turnover and organic matter content in soils, both because they provide C inputs for micrbiological caharacteristics in the soil through litter and exudation in the rhizosphere, and because they stimulate the turnover of existing soil C by rhizosphere microorganisms and their activities (Chen et al.,



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2006). The functional capacity of the soil microbial community, as reflected in the activities of enzymes involved in nutrient mineralization processes, varies among soils dominated by plant roots (Waldrop et al., 2000; Kourtev et al., 2003). Nevertheless, there have been relatively few studies that have examined root exudation, microbial rhizosphere community composition and enzyme activities of plants (Kourtev et al., 2003). Recently, Grierson and Adams (2000) indicate that microbial activity and enzyme activities are strongly affected by plant roots.

Microbial activity plays an important role in regulating soil fertility. Indeed, the microbiological processes taking place in soil are at the centre of many ecological functions (Nannipieri et al., 1990), since microbiological activity is related to soil structure, soil fertility, and the transformation of soil organic matter (Ladd et. al., 1996). Several microbiological parameters have been used to define the status and sustainable development of soil productivity in agricultural ecosystems (Visser and Parkinson, 1992). There are many methods currently available for studying the microorganisms and their activities at the microhabitat level (Nannipieri et al., 1990). The dependence of the microbiological properties of agricultural soils on site and soil factors has been studied (Vekemans et al., 1989). Some soil biological properties such as enzyme activities, respiratory activity and microbial biomass are used as bio-indicators for soil quality and health in environmental soil monitoring (Rogers and Li, 1985).

The microbial biomass, being containing only 1-3% of total soil carbon and approximately 5% total soil (Smith and Paul, 1990), is an important component of soil organic matter. It is involved in biogeochemical cycles of the main nutritive elements (C-N-P-S) and in related energy flows (Meli et al., 2002; Kızılkaya et al., 2004). Basal soil respiration of soil microflora provides useful information on the physiological condition of the pedoecosystem, even though it is a matter some controversy. This respiratory activity takes into account the use of energy by microflora and expresses the efficiency of organic carbon degradation by soil microorganisms (Wardle and Ghani, 1992). As presence of dehydrogenases, which are intracellular to the microbial biomass, is common throughout microbial species and they are rapidly degraded following the cell death, the measurement of microbial dehydrogenase activity (DHA) in soils and sediments has been used extensively, (Bolton et al., 1985; Rossel and Tarradellas, 1991). Therefore, usage of DHA as an index of microbial activity has been suggested (Benefield et al., 1977; Nannipieri et al., 1990; Tabatabai 1994; Kızılkaya and Hepsen, 2004). Urease (UA) is involved in the hydrolysis of urea to carbondioxide and ammonia, which can be assimilated by microbes and plants. It acts on carbon-nitrogen (C-N) bonds other than the peptide linkage (Bremner and Mulvaney, 1978; Kızılkaya and Bayraklı, 2005). Arylsulphatase (ASA) is the enzyme involved in the hydrolsis of arylsulphate esters by fission of the oxygen-sulphur (O-S) bond. This enzyme is believed to be involved in the mineralisation of ester sulphate in soils (Tabatabai, 1994). Also, it may be an indirect indicator of fungi as only fungi (not bacteria) contain ester sulphate, the substrate of arylsulphatase (Bandick and Dick, 1999; Askin and Kızılkaya, 2006; Yertayeva et al., 2019).

The experiment in the present study was conducted in the greenhouse, simulating field conditions of organic matter management with different organic wastes (hazelnut husk, wheat straw, tea waste and tobacco production waste) in soil. The organic wastes used in the research were selected due to their variance in very large interval (C/N; 20 - 171). All organic wastes were sifted from 0.5 mm sieve after grinding in order to eliminate any effect that could be occurred due to magnitude of the particles. Our objectives were to determine the effects of the organic wastes on biological properties such as microbial biomass, basal soil respiration and enzymatic activities (dehydrogenase, urease and arylsulphatase) in rhizosphere and root-free soil.

#### **Material and Methods**

#### Material

#### Soil and organic wastes

Surface soil (0-20 cm) used in this experiment is a Typic Udipsamment and contained 20.60 % clay, 18.36 % silt, and 61.04 % sand. Soil texture can accordingly be classified as sandy clay loam (SCL). The pH in water was 8.1, the oxidizable organic matter content was 1.68 %, and the soil C:N ratio was 13.9. The properties of the organic wastes (Wheat straw (WS), Hazelnut husk (HH), Tea (TEW) production waste and Tobacco (TOW) production waste) was expressed on a moist-free basis and analyzed by standard procedures, given in Ryan et al. (2001).

#### **Experimental procedure**

The soil samples were air-dried in a laboratory and sieved through 0-2 mm screens. The samples (500 g airdried soil) were placed in 600 ml cylindrical plastic container. The organic wastes (WS, HH, TOW and TEW) were thoroughly mixed with the soil at a rate equivalent to 5% on an air-dried weight basis. Then, five individuals of maize (*Zea mays indendata*) seeds were placed in the soils. The moisture contents in the soils were adjusted to 60% water holding capacity (WHC) and the containers were incubated in greenhause for 90 days. The moisture content was maintained throughout the experiment. The maize-planting containers were regarded as rhizosphere and the other containers as root free soil (nonrhizosphere). Changes in the microbiological properties were determined in the root free soil and rhizosphere samples taken in 15, 30, 45, 60, 75 and 90 days after the experiment was conducted. During the sampling of soil the crops were gently pulled out, and the soil remaining on the maize roots was regarded as rhizosphere. At the same time, the root free soil was taken from the nonplanting containers at the same depth. Soil without organic waste addition was used as a control. A randomized complete plot design with three replicates per treatment and soil was used. This greenhouse experiment was total 180 pots. The experiment was performed with the following 10 treatment:

- (1) control for soil (without organic waste addition and plant seed)
- (2) + 50 g kg<sup>-1</sup> hazelnut husk (without plant seed)
- (3) + 50 g kg<sup>-1</sup> wheat straw (without plant seed)
- (4) + 50 g kg<sup>-1</sup> tobacco production waste (without plant seed)
- (5) + 50 g kg<sup>-1</sup> tea production waste (without plant seed)
- (6) control for rhizosphere (without organic waste addition and with plant seed)
- (7) + 50 g kg<sup>-1</sup> hazelnut husk (with plant seed)
- (8) + 50 g kg<sup>-1</sup> wheat straw (with plant seed)
- (9) + 50 g kg<sup>-1</sup> tobacco production waste (with plant seed)
- (10) + 50 g kg<sup>-1</sup> tea production waste (with plant seed)

#### Methods

#### Total organic C and N contents

Total N in soil was determined by digestion and subsequent measurement by the Kjeldahl method (Bremner, 1965). Whole soil samples were sieved through a 150  $\mu$ m mesh to determine total organic carbon by the wet oxidation method (Walkley-Black) with K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>. C/N ratios in soils were calculated as total organic carbon / total nitrogen (Rowell, 1996).

#### **Biological properties in soils and rhizosphere**

All determinations of microbiological properties were performed for the eachsoil sample in triplicate, and all values reported are averages of the three determinations expressed on an oven-dried sample basis at 105  $^{\circ}$ C for 48 h.

#### Microbial biomass carbon and basal soil respiration

Microbial biomass carbon ( $C_{mic}$ ) was determined by the substrate-induced respiration method of by Anderson and Domsch (1978). A moist sample equivalent to 10 g oven-dry soil or cats was amended with a powder mixture containing 40 mg glucose. The  $CO_2$  production rate was measured hourly using the method described by Anderson (1982). The pattern of respiratory response was recorded for 4 h.  $C_{mic}$  was calculated from the maximum initial respiratory response in terms of mg C g<sup>-1</sup> soil as 40.04 mg CO<sub>2</sub> g<sup>-1</sup> + 3,75. Data are expressed as mg C g<sup>-1</sup> dry sample.

Basal soil respiration (BSR) at field capacity (CO<sub>2</sub> production at 22 °C without addition of glucose) was measured, as reported by Anderson (1982); by alkali (Ba(OH)<sub>2</sub>.8H<sub>2</sub>O + BaCI<sub>2</sub>) absorption of the CO<sub>2</sub> produced during the 24h incubation period, followed by titration of the residual OH<sup>-</sup> with standardized hydrochloric acid, after adding three drops of phenolphthalein as an indicator. Data are expressed as  $\mu$ g CO<sub>2</sub>-C g<sup>-1</sup> dry sample.

#### **Enzyme activities**

Dehydrogenase activity (DHA) was determined according to Pepper et al (1995). To 6 g of sample 30 mg glucose, 1 ml of 3% TTC (2,3,5-triphenyltetrazoliumchlorid) solution and 2.5 ml pure water were added and the samples were incubated for 24 h at 37°C. The formation of TPF (1,3,5 triphenylformazan) was determined spectrophotometrically at 485 nm and results were expressed as  $\mu$ g TPF g<sup>-1</sup> dry sample.

Urease activity (UA) was measured by the method of Hoffmann and Teicher (1961). 0.25 ml toluene, 0.75 ml citrate buffer (pH, 6.7) and 1 ml of urea substrate solution were added to the 1 g sample and the samples

were incubated. The formation of ammonium was determined spectrophotometrically at 578 nm and results were expressed as µg N g<sup>-1</sup> dry sample.

Arylsulphatase activity (ASA) was measured according to Tabatabai and Bremner (1970). 0.25 ml toluene, 4 ml acetate buffer (pH, 5.5) and 1 ml of 0.115 M p-nitrophenyl sulphate (potassium salt) solution were added to the 1 g sample and the samples were incubated for 1 h at  $37^{\circ}$ C. The formation of *p*-nitrophenol (*p*-NP) was determined spectrophotometrically 410 nm and results were expressed as  $\mu g p$ -NP g<sup>-1</sup> dry sample.

#### **Statistical Analysis**

All data were analyzed using SPSS 11.0 statistical software (SPSS Inc.). Analysis of variance (ANOVA) was carried out using three factors (plant root, incubation period, organic waste) randomized complete plot design; where significant *F*-values were obtained, differences between individual means were tested using the LSD (Least Significant Difference) test, with a significance level of P<0.01. The asterisks, \*, \*\* and \*\*\* indicate significant at *P*<0.05, 0.01 and 0.001 respectively.

#### Results

#### **Composition of organic wastes**

Among the OW used in this study, TEW had the highest organic matter (92.72%) while that of TOW was the lowest (66.21%). Regarding N content, TEW again had the highest N content (2.46%) and the lowest N content belong to WS (0.31%). C:N ratio of the OW ranged from 20 to 171 and the highest level C:N ratio observed in WS while that of lowest is TOW. The order of OW associated with C:N ratio was WS> HH> TEW> TOW. In addition these OW contained major important nutrients such as P<sub>2</sub>O<sub>5</sub>, K<sub>2</sub>O, which are agronomically important (Table 1).

•	8				
Organic waste	Organic matter, %	C/N	N (%)	$P_2O_5(\%)$	K <sub>2</sub> O (%)
TEW	92,72	22	2.46	0.48	5.83
TOW	66,21	20	1.97	0.45	4.71
HH	85,34	52	0.96	0.28	5.17
WS	91,17	171	0.31	0.25	4.77

Nutrients and biological properties in rhizosphere and root free soil

The organic carbon and N contents in rhizosphere and root free soils were significantly greater in all organic waste treatments compared to the control soil (P < 0.001). All of the organic waste additions significantly increased total organic C in rhizosphere compared to the root free soil (Figure 1 and 2).



Table 1. Composition of organic wastes in measured variables



Figure 1. Changes of total nitrogen in rhizosphere and root free soil. Vertical bars are standard errors.

HH = Hazelnut husk, WS = Wheat straw, TOW = Tobacco production waste, TEW = Tea production waste

Figure 2. Changes of total organic carbon in rhizosphere and root free soil. Vertical bars are standard errors. HH = Hazelnut husk, WS = Wheat straw, TOW = Tobacco production waste, TEW = Tea production waste

The effects of different organic waste treatments on biological properties in rhizosphere and root free soils are presented in Table 2-6. Considerable variations in all biological properties, BSR, Cmic, DHA, UA and ASA were found for the different organic wastes and with/without plant roots at different sampling times. Statistically significant variations were found in all biological properties at various organic waste application and sampling times (Table 7 and 8). Biological properties were also affected by incubation period, organic waste and plant root. The analysis of variance of the results obtained in our experiment on the periodic

sampling times with organic waste showed that all factors (organic waste types, plant roots and incubation periods) significantly influenced all biological properties. After organic waste addition a rapid and significant increase in biological properties was observed in waste amended soils followed by a progressive increase in the biological properties in rhizosphere amended with the organic waste. At the end of the experiment, the biological properties measured in waste-treated soils were statistically different from those measured in the control soils.

Table 2. Microbial biomass C (Cmic) in rhizosphere and root free soils ( $\mu g \ CO_2$ -C g<sup>-1</sup> dry soil). Standard error in parenthesis.

Incubation			Organic wastes		
Days	Control	TEW	TOW	HH	WS
			Rhizosphere soil		
15 days	2,7 (0,43)	8,4 (0,40)	7,5 (0,98)	6,2 (0,24)	5,6 (0,15)
30 days	3,6 (0,11)	12,2 (0,80)	10,3 (0,16)	8,2 (0,14)	6,2 (0,13)
45 days	4,5 (0,09)	15,5 (0,52)	14,7 (0,36)	8,2 (0,30)	8,9 (0,84)
60 days	5,5 (0,35)	18,3 (0,24)	16,2 (0,82)	13,7 (0,61)	9,9 (0,56)
75 days	7,1 (0,14)	20,8 (0,25)	18,4 (1,27)	15,4 (0,34)	12,8 (0,43)
90 days	9,6 (0,70)	20,9 (0,76)	18,3 (0,53)	17,2 (0,37)	15,2 (0,31)
			Root free soil		
15 days	2,6 (0,37)	8,5 (0,18)	7,8 (0,85)	5,8 (0,22)	4,4 (0,41)
30 days	2,5 (0,16)	11,5 (0,16)	9,6 (0,19)	7,4 (0,39)	5,5 (0,38)
45 days	2,8 (0,16)	12,7 (0,57)	10,5 (0,74)	9,4 (0,74)	7,8 (0,56)
60 days	2,9 (0,61)	13,6 (0,49)	11,4 (0,29)	10,1 (0,08)	8,9 (0,68)
75 days	3,0 (0,48)	13,7 (0,49)	12,7 (0,29)	12,5 (0,37)	9,1 (0,11)
90 days	3,1 (0,22)	15,1 (0,42)	13,2 (0,80	13,1 (0,27)	11,0 (0,75)

Table 3. Basal soil respiration (BSR) in rhizosphere and root free soils ( $\mu g \ CO_2 \ g^{-1} \ dry \ soil$ ). Standard error in parenthesis.

Incubation					Organic	wastes					
Days	Con	trol	TE	W	TO	TOW H			H WS		
					Rhizospl	here soil					
15 days	27.5	(4.40)	85.7	(4.12)	29.1	(3.83)	63.9	(2.43)	57.2	(1.53)	
30 days	40.1	(1.22)	137.8	(8.97)	116.1	(1.84)	92.2	(1.57)	69.6	(1.49)	
45 days	44.4	(0.91)	153.0	(5.10)	144.9	(3.89)	92.2	(2.94)	57.8	(4.31)	
60 days	38.3	(2.95)	182.0	(2.95)	161.8	(8.19)	136.5	(6.10)	98.5	(5.56)	
75 days	49.9	(5.81)	212.0	(2.50)	134.7	(8.84)	156.5	(3.48)	130.7	(4.39)	
90 days	101.7	(7.44)	212.7	(8.10)	194.8	(5.62)	182.5	(3.91)	161.3	(3.25)	
					Root fr	ee soil					
15 days	17.3	(1.66)	86.8	(1.88)	52.9	(3.89)	42.5	(3.24)	45.6	(4.21)	
30 days	28.6	(1.80)	129.8	(1.79)	108.6	(2.14)	83.1	(4.40)	41.6	(3.13)	
45 days	18.8	(4.29)	124.9	(5.65)	74.1	(5.74)	92.9	(5.74)	51.7	(3.89)	
60 days	28.4	(6.13)	135.4	(4.92)	113.2	(2.90)	100.9	(0.75)	62.4	(4.83)	
75 days	30.3	(4.93)	139.6	(4.92)	129.7	(2.90)	127.6	(3.75)	93.1	(1.10)	
90 days	33.4	(2.33)	160.6	(4.50)	140.0	(8.51)	139.6	(2.91)	116.5	(8.01)	

#### Discussion

#### Total organic carbon and nitrogen

Total organic C contents in rhizosphere were higher than in root free soil at all organic waste applications (Figure 1). Treatments of TEW and WS gave the highest organic C content in rhizosphere and root free soil compared to the control treatment. In addition, N contents in TOW and TEW treated soils in rhizosphere were significantly greater in all organic waste treatments compared to the control treatment and root free soil. Total N in root free soil were higher than in rhisophere at all treatments. These situations might be related organic matter and N contents of organic wastes which contain different amounts of organic matter and N (Table 1) and N uptake by plant roots. The differences of C/N ratios of rhizosphere and root free soil were statistically significant for all OW treatments. The TOW and TEW treatments had lower C/N ratio in rhizosphere and root free soil than those in other treatments (HH and WS) (Figure 1). All these changes mostly depended on the characteristics and initial level of organic C and N contents of organic wastes. In general, C/N ratios in rhizosphere and root free soil were lower in soil treated with organic wastes of initial low C/N ratios (TOW and TEW), while treatments with high initial C/N ratios (WS and HH) caused high C/N

ratios in rhizosphere and root free soil. Figure 1 shows that the organic C in rhizosphere were higher than in control treatment and in root free soils at all sampling times and organic waste treatments. This situation might be related supply of organic C material from plant exudates such as polysaccharides, mucigel, carbohydrates and amino acids, and dead cells of root hairs (McGill et al., 1986; Huang and Schoenau, 1998). Table 4. Dehydrogenase activity (DHA) in rhizosphere and root free soils (µg TPF g<sup>-1</sup> dry soil). Standard error in

Table 4. Dehydrogenase activity (DHA) in rhizosphere and root free soils (µg TPF g<sup>-1</sup> dry soil). Standard error in parenthesis.

Incubation			Organic wastes		
Days	Control	TEW	TOW	HH	WS
			Rhizosphere soil		
15 days	16,9 (0,65)	87,2 (3,40)	76,9 (2,31)	46,2 (1,83)	22,5 (1,43)
30 days	20,8 (0,69)	103,7 (5,96)	80,2 (2,12)	55,5 (2,67)	27,4 (2,94)
45 days	24,5 (0,59)	110,2 (1,90)	96,4 (3,07)	55,5 (2,25)	49,6 (12,52)
60 days	27,9 (2,34)	137,0 (3,56)	116,0 (5,46)	75,7 (4,03)	50,3 (2,39)
75 days	32,4 (2,14)	156,5 (5,68)	138,6 (1,85)	96,1 (4,08)	69,2 (3,24)
90 days	39,9 (2,79)	193,0 (8,73)	171,3 (1,74)	118,0 (10,04)	78,7 (2,66)
			Root free soil		
15 days	14,9 (0,60)	80,4 (1,84)	69,0 (1,18)	39,9 (2,06)	20,1 (0,93)
30 days	14,3 (0,80)	84,5 (3,56)	73,2 (2,18)	45,3 (1,29)	21,9 (1,63)
45 days	15,3 (0,91)	99,3 (3,70)	91,0 (2,75)	58,6 (2,75)	25,5 (1,46)
60 days	15,9 (0,32)	111,2 (3,66)	98,2 (2,81)	60,4 (3,02)	31,3 (2,14)
75 days	15,9 (1,36)	132,5 (3,66)	112,6 (2,81)	79,2 (3,51)	50,0 (2,29)
90 days	16,3 (0,94)	138,6 (8,40)	127,9 (4,00)	87,9 (4,89)	66,1 (7,78)

Table 5. Urease activity (UA) in rhizosphere and root free soils ( $\mu$ g N g<sup>-1</sup>dry soil). Standard error in parenthesis.

Incubation			Organic wastes			
Days	Control TEW		TOW	HH	WS	
			Rhizosphere soil			
15 days	7,0 (0,54)	8,7 (1,06)	16,7 (2,54)	8,0 (0,75)	7,6 (1,17)	
30 days	17,2 (2,29)	34,0 (1,95)	32,4 (0,74)	26,1 (1,79)	21,5 (0,92)	
45 days	21,8 (1,93)	38,7 (1,69)	39,4 (1,18)	26,1 (2,33)	26,4 (2,09)	
60 days	25,6 (1,24)	42,8 (2,24)	46,2 (2,74)	32,3 (1,93)	30,5 (2,14)	
75 days	32,4 (2,14)	58,2 (3,02)	58,6 (1,85)	42,8 (2,27)	40,2 (1,53)	
90 days	32,9 (2,70)	74,7 (3,09)	71,3 (1,74)	58,6 (3,01)	44,3 (2,73)	
			Root free soil			
15 days	5,4 (1,18)	9,1 (1,57)	13,5 (2,85)	8,9 (0,84)	7,1 (1,59)	
30 days	14,2 (2,30)	27,2 (1,31)	27,8 (2,91)	23,7 (2,15)	19,2 (1,00)	
45 days	13,5 (1,90)	34,3 (1,31)	33,0 (1,27)	25,3 (1,27)	21,7 (1,86)	
60 days	15,9 (0,32)	38,4 (2,19)	39,6 (0,96)	29,8 (1,69)	26,5 (1,98)	
75 days	15,9 (1,36)	38,9 (2,19)	39,6 (0,96)	32,8 (2,69)	30,0 (2,29)	
90 days	19,1 (1,24)	58,6 (2,70)	52,7 (2,84)	40,9 (2,33)	35,6 (0,97)	

Table 6. Arylsulphatase activity (UA) in rhizosphere and root free soils ( $\mu g \text{ p-NF } g^{-1} \text{ dry soil}$ ). Standard error in parenthesis.

Incubation					Organic	c wastes				
Days	Control		TE	TEW		TOW		HH		/S
					Rhizosp	here soil				
15 days	22,7	(2,12)	84,4	(4,34)	67,4	(1,90)	47,1	(1,42)	31,1	(1,03)
30 days	28,8	(2,81)	87,3	(3,94)	81,2	(2,79)	59,3	(4,35)	35,0	(2,44)
45 days	31,9	(1,81)	103,1	(4,39)	96,0	(5,13)	59,3	(1,45)	41,4	(3,33)
60 days	39,5	(1,26)	122,3	(2,82)	105,1	(4,96)	84,6	(2,78)	62,4	(3,61)
75 days	45,6	(1,98)	165,0	(9,58)	134,3	(7,86)	97,8	(3,06)	74,5	(2,66)
90 days	58,1	(1,98)	180,4	(10,83)	151,5	(5,50)	129,6	(8,26)	92,5	(2,85
					Root fi	ree soil				
15 days	10,7	(1,34)	38,9	(1,80)	30,8	(1,20)	26,9	(2,97)	17,3	(2,04)
30 days	13,4	(1,01)	61,3	(4,35)	59,5	(2,18)	36,5	(2,99)	19,2	(1,00)
45 days	15,7	(1,18)	74,9	(4,29)	63,0	(1,27)	43,6	(1,27)	33,4	(3,70)
60 days	20,5	(1,30)	94,7	(3,61)	82,1	(2,95)	59,0	(1,91)	44,4	(2,73)
75 days	20,6	(0,97)	124,6	(3,61)	105,7	(2,95)	74,9	(4,16)	73,6	(2,48)
90 days	25,52	(1,01)	141,6	(8,04)	122,0	(4,47)	96,9	(2, 37)	66,6	(2,78)

Variables	BSR		Cmic			
	F-value	$LSD_{\alpha=0.01}$	F-value	$LSD_{\alpha=0.01}$		
Plant root (Pr)	73.210***	8.516	1272.261***	0.200		
Incubation days (Id)	67.507***	14.749	981.591***	0.347		
Pr x Id	5.060***	20.859	117.093***	0.490		
Organic wastes (Ow)	128.453***	13.464	2099.943***	0.316		
Pr x Ow	0.435***	19.041	17.559***	0.447		
Id x Ow	2.913***	32.981	25.607***	0.775		
Pr x Id x Ow	1.217***	46.642	6.615***	1.096		

#### Table 7. Results of ANOVA for BSR and Cmic

Table 8. Results of ANOVA for enzyme activities

Variables	DHA		UA		ASA	ASA		
	F-value	$LSD_{\alpha=0.01}$	F-value	$LSD_{\alpha=0.01}$	F-value	$LSD_{\alpha=0.01}$		
Plant root (Pr)	699.030***	1.558	684.391***	0.774	1843.665***	1.492		
Incubation days (Id)	851.059***	2.751	1393.715***	1.341	1436.351***	2.584		
Pr x Id	46.014***	3.890	67.705***	1.897	8.664***	2.359		
Organic wastes (Ow)	3688.078***	2.511	713.288***	1.224	2484.417***	3.665		
Pr x Ow	12.647***	3.551	8.349***	1.731	39.071***	3.336		
Id x Ow	42.717***	6.150	35.649***	2.999	57.859***	5.778		
Pr x Id x Ow	5.413***	8.698	2.475***	4.241	4.246***	8.172		

#### **Biological properties**

Different organic waste application significantly affected the levels of biological properties in the rhizosphere, when compared with the control treatment and root free soils. Table 2-6 shows that the BSR, Cmic and enzyme activities (DHA, UA and ASA) in rhizosphere were higher than in control treatment and in root free soils at all sampling times and organic waste treatments. This situation might be related the supply of organic material from plant roots and plant exudates. The supply of organic material from plant roots is crucial to soil microbial communities whose growth is carbon limited. The type and amount of nutrients released will affect both the microbial biomass and their activity. This primary carbon supply to the soil system arrives through plant litter and more directly from roots. These include the release of plant exudates, many of which appear to be simply lost by leakage from the root. Plant exudates contain carbohydrates, amino acids, organic acids, lipids, hormones, vitamins and enzymes. These organic substances are stimulated for soil microbiological activity. It is well known that root-derived organic C from root exudates stimulates the growth of microorganisms and increases microbial activity in the rhizosphere (Toal et al., 2000; Kourtey et al., 2003; Bais et al., 2004). Results from this study also showed the greater biological properties (Cmic, BSR, and enzyme activities such as DHA, UA and ASA) in all organic waste added soils under plant roots compared with root-free soil. Greater biological properties in all organic waste added soils under rhizosphere after 90 days contributed to greater under root free soil. It is likely that increased levels of organic C and N due to root exudation could have led to greater microbial activity. The amount of rootderived C flow through the rhizosphere has a significant impact on transformations of soil organic C, N, P and S (Helal and Sauerbeck, 1989). It has been established that soluble organic C and N in mineral soils is mainly derived from root derived from root exudates and root residues (McGill et al., 1986; Huang and Schoenau, 1998). In the present study, organic C accumulated in the rhizosphere soil, and concentrations of organic C were significantly greater in the rhizosphere compared with root free soil. There were significant relationships observed between biological properties and organic substrates in rhizosphere and root-free soil, indicating that greater value of biological properties such as Cmic, BSR and enzyme activities in the rhizosphere may be partly attributed to increased levels of organic C. It has been suggested that both plant roots and microorganisms produce UA and ASA (Speir and Ross, 1978). It was found that UA and ASA were higher in the rhizosphere compared with control and root-free soil (Table 5 and 6), which is consistent with findings from several other studies (Tarafdar and Jungk, 1987). Moreover, UA and ASA were directly related to value of microbial biomass and their activities in soils.

These tables shows that both rhizosphere and root free soils BSR, Cmic and enzyme activities (DHA, UA and ASA) in all organic waste treatments were higher than in control treatment at all sampling times. This situation may be related carbon source of organic wastes and increased the organic matter level, which consequently elevated the biological properties of soil. For this reason, increased soil organic matter content is correlated positivitely with microbiological activity in soil, generally. The organic waste treatments had consistent or significant effect on the soil biological properties. This indicated accumulation of organic matter and improvement in nutrient status of soil, as microbial biomass and their activity is a labile

reservoir of plant nutrients (Jenkinson and Ladd, 1981). The BSR, Cmic and enzyme activities (DHA, UA and ASA) in rhizosphere and root free soil for all organic waste treatments was similar in all sampling times (Table 2-6). Addition of organic material increases the microbial activity in soil (Pascual et al., 1997). García-Gil et al. (2000) reported increases microbial biomass and their activity in soil organic waste application application to soil. Such increases in rhizosphere and root free soil BSR and Cmic were probably caused by the higher level of soluble organic-C in organic wastes. Availability of biogenic material for biomass stimulation (Jenkinson and Ladd, 1981) induced the increase in soil microbial activity of enriched soils. The increase may also correspond to the growth of the zymogenous population associated with organic matter enrichment (Jenkinson et al., 1987) and incorporation of exogenous microorganisms (Perucci, 1992). The source of enzymes in soil is definitely known, additionally presumed to originate from microorganisms, plant roots and soil animals (Tabatabai, 1994). However, evidence could be obtained from the present study that DHA, UA and ASA of root free soil and rhizosphere were positively related to Cmic and their activity. Perucci (1992) also found positive correlation between enzyme activities and microbial activity. Addition of all organic wastes increased the enzyme activities of rhizosphere and root free soil. This could have originated from the higher amounts of enzymes in the viable microbial populations and the increased levels of accumulated extracellular enzymes (UA and ASA) in the soil matrix. Presence of enzymes in organic matter (Dick and Tabatabai, 1984) may also contribute enzymes directly to soil on addition.

The highest BSR, Cmic and enzyme activities (DHA, UA and ASA) were generally found in rhizosphere and root free soil at TOW and TEW treatments. There have been numerous studies (Pascual et al. 1997; Madejón et al., 2001) on the effects of organic wastes on microbial activity and their enzymatic activities in soil. These studies generally indicated larger effects in organic matter or organic waste treated soils than in control or non-treated soils. However, in most studies it was possible to establish relationships between and biological properties and the magnitude of the effects of organic waste type's especially chemical composition, nutrient content and C/N ratio. Similarly, Martens et al. (1992) suggested that variation in the nature of organic materials variably stimulated the microbial activity and production of enzyme activity in soil. In this researh, higher enzyme activities (DHA, UA and ASA) of soil treated with TOW and TEW was associated with their quality in respect to their capacity of microbial biomass production. This situation might be related initial C/N ratios of organic wastes. Organic wastes their C/N ratios are the most important factors that the effects on soil biological properties. Moreover, low C:N ratio and nutrient (N,P,K) sources are essential for the buildup of Cmic and the production or synthesis of enzymes (Alexander, 1977). This can obviously be explained by the input of nutrients in organic wastes and lower C/N ratios prevalent in TEW and TOW. This rose with organic waste, particularly when TOW and TEW were added since this contains a high proportion of easily biodegradable compounds compared with HH and WS. Nitrogen content is also important in determining microbial decomposition rates of organic waste. Higher decay rates are found with increased nitrogen supply (Marinucci et al., 1983). This is similar to the role played by nitrogen in decomposition of other types of organic matter (Mann, 1976; Berg et al., 1982). In addition, nitrogen content of organic wastes has only a positive effect on decomposition rates (Valiela et al., 1985). Because nitrogen has a positive effect on decomposition rates, the trend of increasing nitrogen content within decomposing wastes during the microbially-dominated stage of decomposition is important.

#### Conclusion

According to data, this showed a clear relationship between organic wastes and biological properties. We assume that the replacement of organic waste has stimulating effects on biological properties such as Cmic, BSR and enzyme activities (DHA, UA and ASA) in rhizosphere and root free soil, due to the quantity and quality of the organic waste incorporated into soil, and the microbial growth caused by the addition of organic compounds to the soil. Organic materials are possibly the most important C source for microorganisms. It consists mainly of root exudates and organic waste degradation products. Differing organic waste inputs in the system were reflected by the C and N contents which, however, varied much more between the systems than did biological properties. In general, initial low C/N ratios of organic wastes application (TEW and TOW) caused the most beneficial effects on biological properties in rhizosphere and root free soil among the investigated types of organic waste on clay loam soils. The use of these organic wastes can contribute to an enhancement of the level of organic matter and the fertility of the agricultural soils. Furthermore, organic waste had a stronger impact on biological properties in rhizosphere compared to root free soil. Hence, it can be concluded that the biological properties was clearly governed by the organic waste incorporated into soil under the conditions of the investigated greenhouse experiment. At the same time this practice seems to be a potentially effective way of recycling wastes and solving the problem of their disposal.

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# Seed priming with NaCl improves germination in maize under saline soil conditions

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#### Abstract

Soil salinity is considered crucial for seed germination, seedling growth, and crop production in arid and semi-arid regions. Seed priming can be an effective solution to improve maize germination and growth, under salinity stress. An experiment was conducted to study the effect of seed priming with NaCl and salinity stress on germination and growth of maize variety Arun-2. Before sowing, eighty maize seeds were soaked in 5g/L aqueous NaCl solution for 12 hours at 27-degree Celsius. Primed and unprimed seeds were sown in 10-liter capacity plastic pots and watered with 200mL of 0, 2, 4, 6, and 8 g/L saline solutions at two days intervals. Germination percentage, shoot length, and number of leaves per plant were measured to access the germination and growth parameters. The results showed that priming seeds with NaCl solution significantly (P<0.05) improved the germination of maize seeds. Whereas, germination of Arun-2 was not significantly affected by salinity stress. Salinity has negative impacts on shoot length and number of leaves. The shoot length at 38 DAS was found to be the longest at salinity level 0 mg/L (8.61cm) and it was found to be shortest at highest salinity level i.e. 8 mg/L (3.12 cm). Increasing salt stress has severe effects on the growth of maize, both during the seedling and vegetative growth stages of the plant. Seed priming alleviated the inhibitory effect of salt stress on germination and seedling growth of maize. Thus, seed priming with 5 g/L NaCl solution could be useful to improve the germination and growth of maize under saline stress conditions.

Keywords: Maize, NaCl, salinity, seed germination, seed priming, seed growth.

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#### Introduction

Salinity is one of the crucial environmental factors that limit plant growth and productivity (Kaya et al., 2002). Saline soil contains a huge amount of soluble salts and sodium ions. These ions increase the osmotic potential in the soil which prevents easy absorption of water by the plants (Omuto et al., 2020). Salinity is a severe problem in arid and semi-arid regions of the world with less available water to the plants. Soil salinity

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Publisher : Federation of Eurasian Soil Science Societies e-ISSN : 2147-4249 also causes various physiological disorders in plants. Elevated salinity level in soil affects water uptake, causes nutritional disparity, and inhibit photosynthetic efficiency in plants (Okon, 2019). Soil salinity drastically reduces the yield. These reductions may be due to various physiological disorders and biochemical changes resulted from saline soil.

Maize is a moderately salt-sensitive crop (Carpici et al., 2009). Cultivation of maize under saline soil conditions affects plant germination, plant establishment and ultimately reduces the yield. The development of salt-tolerant maize requires a considerable amount of time and resources. Researchers found that Salt tolerance in maize can be induced by priming maize seeds with chloride salts (Ashraf and Rauf, 2001). Validation of this approach using locally cultivated genotypes could be beneficial to the low scale maize growers working on saline soil conditions.

Seed priming is a common method used in vegetable crops for better germination. It is getting popular and is being used as a successful method to improve seed germination and crop establishment in developing countries. This method is easy to use and cost-effective for resource-poor farmers and farming systems (Harris et al., 2002). Seed priming stimulates various biochemical changes (water imbibition, breaking of dormancy, activation of certain enzymes) in the seed that are essential to initiate the germination process (Ajouri et al., 2004). Several biochemical processes stimulating germination are activated by seed priming and the activation will remain even after re-desiccation of those seeds (Asgedom and Becker, 2001). Most of the seed priming works has been done on vegetable and other field crops, therefore this study was conducted to explore the effects of seed priming on growth parameters of maize plants under salinity stress condition.

Most of the rural populations in Nepal are based on agriculture. About 28 percent of the land is available for agricultural purposes (MoAD, 2019). Cereals are cultivated as major crops that contribute to the national economy and fulfills the dietary requirement of the people (MoAD, 2019). Among the cereal crops, maize is grown mostly across the country after the rice. However, most of the cultivated land in rural parts of Nepal are saline. Due to the sensitive nature of maize to saline soil, the germination and growth of maize under such conditions are greatly affected (Farooq et al., 2015). Advanced breeding programs to develop salt-tolerant varieties and/or use of modern biotechnological tools are well-established options to mitigate salinity stress in agriculture (Prasanna et al., 2021). Yet, the time required to develop salt-tolerant varieties is long. Unfortunately, local farmers are lagging to adopt the new technologies. Seed priming with chloride salts is fast and could be an effective technique to mitigate soil salinity problems in rural areas. The farmers can use their locally adapted germplasm and cope with the challenges of soil salinity. This experiment was conducted with the objective to see the effectiveness of seed priming with NaCl in maize germination and growth under saline soil conditions.

#### **Material and Methods**

#### Study Site and Experimental Design

The study was conducted at the experimental farm of Tribhuvan University, Gokuleshwor Campus, Baitadi Nepal in the summer season (April-June) of 2019. The study site is situated at 29° 40′ 0″ N, 80° 34′ 0″ E and 1600 meter above sea level. The average daily temperature during the research duration was 27 degree Celsius and the average relative humidity was 65 percent. Forty plastic containers (40 cm x 35 cm) were used for the experiment. Twenty pots were allotted to grow the primed seeds and the remaining twenty pots were allotted as unprimed (control) pots. The containers were kept at open environment and drilled at the bottom and the sides for water drainage and aeration, respectively. The bottom of each pot was lined with drainage sand to keep the soil well-drained and was filled with 1:3:1 of sand, local soil (the growth medium), and manure fertilizer, respectively. Ten seeds were sown in each pot at 2 cm depth in quadruplicates. The experiment was conducted with a completely randomized design with four replications. There were two factors in the experiment: salinity level and priming. Five salinity concentrations (0, 2, 4, 6, and 8 g NaCl/L) two methods of priming (primed and unprimed) were used. Each salinity level and priming combinations were randomized among the pots and were also replicated.

#### Priming and salinity treatment

Maize variety 'Arun-2 was selected for the experiment due to short crop duration that is 80 to 90 days and suitable for mid-hills of Nepal. The variety was brought from local agrovet. The seeds were surface sterilized (disinfected) with sodium hypochlorite (NaOCl) solution for 3 minutes and then thoroughly washed for 5 minutes using distilled water. Subsequently, the seeds were primed by soaking with NaCl solution of 5 g/L for 12 hours at room temperature. The ratio of seed weight to NaCl solution was 1:5 (g/mL). After priming,

seeds were removed and washed with tap water and then rinsed three times using distilled water. Finally, the seeds were placed between two filter papers to remove the moisture.

Each salinity level 0, 2, 4, 6, 8g/L) was applied on four pots that contained the primed seeds, and another four pots that contained unprimed seeds. Each pot was irrigated with 200 mL of saline solution at two days intervals. After the 10th day of seedling, plants were thinned to maintain three plants per pot. The number of seeds germinated were counted five days after seeding and converted into percentage germination. Shoot length and number of leaves per plant were measured starting from ten days after seeding and measured at one-week intervals. Shoot length was measured for five consecutive weeks and the number of leaves was measured for three weeks.

The data were analyzed using two-way ANOVA in MSTAT 5.4 software. Mean comparisons were made using least-square means (LSD) tests and significance were detected when the p-values were less than 0.05.

#### Results

The effects of seed priming and salinity concentration on seed germination, shoot growth, and the number of leaves were discussed below.

#### Effect of priming with NaCl on seed germination

Significant improvement in germination percentage was observed in primed seed (95.50) compared to normal seed (78.50) (Figure 1). There were no significant differences in percent germination among the salinity levels (Figure 2). The mean value showed that the maximum germination percent (90%) was observed in salinity level 2g/L and 8g/L, whereas the minimum germination percentage (82.5) was reported when the salinity level was 6g/L.





Figure 1. Effect of seed priming on germination percentage of maize seed. Different letters at the top of the bars represent significant differences at p<0.05.



#### Effect of seed treatment and salinity level on shoot length

Shoot lengths were measured every week from the 10<sup>th</sup> day of seeding. Analysis of variance showed significant days after seeding (DAS) and salinity level interaction. Therefore, mean comparisons among the salinity levels were conducted separately for each day after seeding (DAS). No significant mean differences in shoot length were observed among the salinity levels at 10 days after seeding (Figure 3). However, salinity level 6g/L showed the maximum shoot length (2.18 cm). There were significant differences in mean shoot length among different salinity levels at 17, 24, 31, and 38 days after seeding. The mean shoot length showed a decreasing trend as the salinity level increases. Furthermore, there were significant differences in shoot length when the salinity level was 0 compared to any other salinity level (Figure 4). Shoot length at salinity level 2g/L was different from salinity level 8g/L at 17, 25, 31, and 38 DAS. Shoot length at salinity level of 2g/L did not differ from shoot length at salinity level 4g/L. Similarly, shoot length at salinity level 4g/L did not differ from the shoot length at 6g/L at 17, 24, and 38 DAS. However, they differed at 31 DAS. Overall analysis indicated that shoot length is higher when there is no salinity in the soil. No differences between the mild salinity levels (2g/L and 4g/L) were observed, but higher salinity (8g/L) drastically reduced the shoot length in maize. There were no significant differences in shoot length between the primed and unprimed seeds (Figure 5). However, the means of unprimed seeds were lower at all the days after seeding.

#### Effect of seed priming with Nacl on leaf number

There were significant differences in the number of leaves among the salinity levels at 10, 17, and 24 days after sowing (Figure 6). On days 17 and 24, the mean number of leaves at salinity level 0 was different from

the mean number of leaves at any other salinity level. At 10 days after sowing the number of leaves per plant at 2g/L was different from 0g/L, but not different from salinity level 4, 6, and 8g/L. This indicated, during the initial days of plant growth, salt stress did not have a significant effect on number of leaves. At 17 DAS increasing the salt stress reduced the number of leaves in plants. Data also showed that there were differences in the number of leaves per plant when salt stress was moderate (2g/L and 4g/L) compared to high salt stress (6g/L and 8g/L). Salinity stress drastically reduced the number of leaves at 24 DAS. The reduction in leaf number was more when the salinity stress was increased (Figure 7). There were no significant differences in leaf number between primed and unprimed seeds (Figure 8). However, the mean value indicated that the leaf numbers were more in primed seeds compared to unprimed seeds.





Figure 3. Effect of salinity on shoot length of maize seedling. The same letters at the top of the bars represent no significant differences. Different letters indicate significant differences at P<0.05.

Figure 4. Effect of salinity on shoot length of maize seedling. 0g/L indicates no salt treatment. The shoot length is negatively affected by salinity stress compared to non-stressed.





#### Discussion

Seed priming is one of the effective methods to mitigate salinity stress in plants (Imran et al., 2018). Various priming techniques have been suggested by many researchers. Polyethylene glycol (PEG), water, chloride salt of sodium, potassium, and calcium are some of the priming agents used in seed priming (Ashraf and Rauf, 2001). No standard priming agent was found to be clearly better than the other. Seed priming stimulates various metabolic and physiological processes in plants during germination and early growth (Abraha and Yohannes, 2013). The present study investigated the effect of seed priming and salinity stress on the germination and growth of maize. Sodium chloride solution was used as a salt to prime maize seeds before sowing. Salinity did not have a significant effect on the germination of maize seeds. There were significant differences in germination percentage between primed and unprimed seeds. Higher germination percentage was observed in primed seeds compared to the unprimed ones. One of the reasons for better germination of primed seeds in stress conditions was due to the improvement in water imbibition (Chen et al., 2021). Seed priming with sodium salt was also found to be increase the water use efficiency in maize (El-

Sanatawy et al., 2021). Besides imbibition, pre-sowing treatment of maize seeds with sodium salt may have initiated the early metabolic process, activated various enzymes, and enhanced physiological activities inside the seed (Marthandan et al., 2020). There were no statistically significant differences in shoot length and number of leaves between primed and unprimed seeds. However, it has been reported that better seedling growth and vigor in primed seeds compared to the unprimed ones. Chen et al. (2021) found that seed priming helped to reduce salt stress damage and also improved seed germination and vigor. They further found, seed priming promoted a strong establishment of seedling and has better root and shoot traits. No significant differences between primed and unprimed seeds for shoot length and number of leaves were observed. This may be due to higher variation in our experimental data. It may also be due to lower seed weight: NaCl ratio or due to a varietal response of Arun-2. However, in all the cases the mean values of shoot length and number of leaves were higher in primed seeds compared to the unprimed ones.

**Number of leaves/Plant** 



Figure 6. Effect of salinity on number of leaves/Plant of maize seedling. The same letters at the top of the bars represent no significant differences. Different letters indicate significant differences at P<0.05.



Figure 7. Effect of salinity on number of leaves/Plant of maize seedling. 0g/L indicates no salt treatment. The number of leaves/Plant is negatively affected by salinity stress compared to non-stressed.



Figure 8. Effect of seed priming on number of leaves/Plant of maize seedling. The same letters at the top of the bars represent no significant differences.

Salinity stress significantly reduced the shoot length and leaf number in maize. Even the mild salinity stress significantly reduced the shoot length compared to the non-stressed condition. The shoot length decreased drastically as the salinity level increased. Similarly, the number of leaves also decreased with the increase in salinity stress. Reduction in seedling growth with increased salinity level was also found by Tsegay and Andargie (2018). This reduction was due to the decrease in water uptake capacity of the seedlings. Alterations in various physiological activities such as change in enzyme activity, reduced sink activity, reduction in stomatal conductance, and reduction in photosynthetic activity were responsible for reduced shoot growth in saline soil conditions (Chutipaijit et al., 2011). Higher salinity concentration had severe

effects on plant growth. Apart from distortion in plant growth, in some cases, plants do not even reach the reproductive stage (Bakht et al., 2011). The reduction in number of leaves with increased salinity stress may also have negative effects on the photosynthetic activity of the plants. This ultimately affects the proper growth and development of plants.

#### Conclusion

Salinity inhibits germination and seedling growth in maize. Seed priming is an effective method to increase salinity tolerance in maize fields. The better performance of primed seeds in this experiment illustrates the necessity of priming seeds before sowing in the saline soils. Seed priming induces germination and helps in the overall growth and development of the plant. Salt sensitive nature of maize variety Arun-2 makes it unsuitable to grow in saline soil without seed priming. Although our research found the usefulness of seed priming in the early growth stages of maize, additional research on late growth and reproductive stages would be beneficial. Advanced research on priming induced alteration of physiological and biochemical attributes in maize will be the goal of our future research.

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## Influence of the biochar on petroleum hydrocarbon degradation intensity and ecological condition of Haplic Chernozem

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#### Abstract

It was evaluated the impact of biochar on the petroleum hydrocarbons degradation intensity and the ecological condition of Haplic Chernozem. The study was conducted in the model experiment conditions with Haplic Chernozem contaminated with 5% of petroleum hydrocarbons with application of 10 and 20% biochar. The number of soil bacteria, the activity of catalase and dehydrogenases, germination ability and radish root and shoot length, soil respiration considered to evaluate biological activity. It has been established that 10% biochar application led to intensification of petroleum hydrocarbons degradation up to 17% compare to contaminated soil. Upon adding 10% biochar the CO<sub>2</sub> emission increased up to 70-85% on the 18-19th days, then reduced by the 28-30<sup>th</sup> days till soil emission with the application of biochar in the amount of 20% from soil mass. Activity of dehydrogenases of Haplic Chernozem were stimulated after application of 10% biochar up to 49% compared to control. Biochar application in the doses of 10 and 20% increased the number of soil bacteria up to 209 and 203%, respectively. Application of 20% biochar intensified the germination and early growth of radish seeds. The germination ability, length of radish shoots and roots increased to 44, 66 and 44%, respectively, compare to control. The biochar application in petroleum contaminated soil increased the activity of catalase, dehydrogenases and the number of soil bacteria. Biochar using in doses 10% and 20% contributes to acceleration of petroleum hydrocarbons degradation and improvement of the soil ecological condition.

**Keywords**: Biochar, petroleum hydrocarbons contamination, soil, bioremediation, residual content of petroleum hydrocarbons

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#### Introduction

Soil and agricultural land contamination with petroleum hydrocarbons constantly increases at the growth of petroleum hydrocarbons consumption. According to the data of the Ministry of Energy of Russia, the production of petroleum hydrocarbons is annually over 550·109 kg (ME, 2021). At the present time, sales and petroleum hydrocarbons export into the commonwealth of independent states (CIS) and non-CIS countries have increased by more than 240·106 ton every year (Neftegaz, 2021). In the case of petroleum hydrocarbons ingress into the soil, all physical, biological, and ecological functions of this soil are violated. According to the concept "Strategic Directions for Stable Social and Economic Development of Agroindustrial Complex of Russia" presented in the Presidium of the Russian Academy of Sciences in 2017, the fundamental change is required in land relation system and production ecological and adaptation to climate changes (Ushachev, 2017). The climate change due to greenhouse gas emission officially executed thanks to concept of "4 ppm", carbon retention and its sequestering for the soils of Russia and the world as a whole are required. At anthropogenic load, for example, at spillages of petroleum hydrocarbons, the introduced carbon



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Publisher : Federation of Eurasian Soil Science Societies e-ISSN : 2147-4249 disturbs the balance in the natural ecological system. The up-to-date methods of petroleum hydrocarbons contaminated soil detoxification consist of acceleration in petroleum hydrocarbons degradation in the soil with soil fertility restoration and quality (Volchko et al., 2013; Zhang et al., 2016; Al-mansoory et al., 2017).

Applying organic fertilizers changes the texture, structure, size distribution of pores and density, gas exchange, water-retaining capacity, plant growth, and soil structure (Downie et al., 2009; Lu et al., 2010; Popova et al., 2019; Abbaspour et al., 2020). Carbon sequestering and contaminant immobilization using the organic absorbing material (activated carbon, biochar and carbon nanotubes) is restored of the contaminated soils (Wang et al., 2018). Compared with other carbon-bearing materials, biochar as a sorbent has shown the best results to provide the possibility of its use to restore the contaminated sediments of soil. For example, the cost of activated carbon is about 10 USD·m<sup>-2</sup>, while the cost of biochar is about 2 USD·m<sup>-2</sup>. Biochar as a main restorative agent is widely used for removal and decontamination of organic compounds, heavy metals and inorganic contaminants ( $NH_4$ ,  $PO_4$  and  $NO_3$ ) in wastewaters, as well as a soil additive for immobilization or isolation of organic contaminants, heavy metals (Ye et al., 2017; Yin et al., 2017; Wang, Wang, 2019). Biochar is obtained by biomass pyrolysis (agricultural wastes, wood wastes and wastewater mud) under anaerobic condition. The physical and chemical properties of biochar significantly influence its capability to immobilize contaminants from sediments and soils (Suliman et al., 2016). In biochar, the functional groups are represented by carboxyl (COOH), hydroxyl groups (OH), amine groups (-NH<sup>4</sup> +) and aromatic compounds (-C=C-), which determine interaction mechanisms between biochar and contaminants in bottom sittings (Hung et al., 2020). Pyrolysis temperature and raw biochar biomass structure are the main factors associated with the functional groups (Leng and Huang, 2018). Availability of such groups allows ensuring higher immobilization capacity of biochar in the bottom sittings (Suliman et al., 2016). Biochar's influence on the ecological soil condition at different types of contamination has been studied insufficiently (García-Delgado et al., 2015; Malyan et al., 2019; Soudejani et al., 2019; Abbaspour et al., 2020).

The work objective is an evaluation of the influence of biochar on petroleum hydrocarbon degradation intensity and the ecological condition of Haplic Chernozem Calcic.

#### **Material and Methods**

#### Soil samples

Haplic Chernozem Calcic was selected as the subject of the research. The sampling place is arable soil (top layer- 0-10 cm) of the Botanical Garden of the Southern Federal University, Rostov on Done (sampling point: 47°14'17.54"N 39°38'33.22 "E). Haplic Chernozem Calcic in the south of Russia is the most fertile soil in Russia. Restoration and retention of soils after anthropogenic load are especially relevant. For the restoration of petroleum hydrocarbons contaminated Haplic Chernozem Calcic functions by petroleum hydrocarbon degradation, the organic carbon-bearing fertilizer - biochar - was used.

#### **Biochar preparation**

Biochar is pure charcoal produced during pyrolysis of birch wood (A grade), with carbon content of at least 85% (GOST 7657-84, 1984). The product is made by birchwood (*Betula alba*) pyrolysis at temperature of 360-380°C without access of oxygen by retort units. Biochar was applied based on 10 and 20% to the mass of the soil.

#### **Modelling of experiment**

After sampling, the prepared common Haplic Chernozem Calcic was dried, sorted out for plant root removal and screened through a metal sieve with a diameter of a mesh of 2 mm. Soil samples was placed in vegetative pots (mass of soil 200 g) in triplicate. Petroleum hydrocarbon with a concentration of 5% of the soil mass (w/w) was applied to moistened soil (up to 60% from soil mass, (w/w)). Soil moisture content was kept within the whole period of incubation. The experiment scheme is given in Figure 1.



Figure 1. Scheme of the experiment for the introduction of biochar concentration of 10 and 20% into non-contaminated and into the petroleum carbohydrates-contaminated Haplic Chernozem Calcic Note: PHC, petroleum hydrocarbon

The petroleum hydrocarbon was kindly provided by Novoshakhtinsk's Refinery (Novoshakhtinsk, Rostov region) and used for contamination modelling. This petroleum hydrocarbon was characterized as light petroleum hydrocarbon (density of 0.818 g/m<sup>3</sup>). Other properties were summarized as follows: the sulfur mass fraction of 0.43%, mechanical impurities mass fraction of 0.0028%, water mass fraction of 0.03%, at chloride salt concentration of 40.1 mg/dm<sup>3</sup>.

#### Indicators of ecological condition

Upon completion of petroleum hydrocarbon contaminated soil and bioremediation agent exposition residual content of petroleum hydrocarbon and the number of biological indices were determined characterizing the ecological condition of the soil after bioremediation: activity of dehydrogenases and catalase, phytotoxic indices, soil respiration (CO2 emission), the number of soil bacteria (Kazeev et al., 2016).

The residual petroleum hydrocarbon content was determined by the infrared spectrometry method using carbon tetrachloride as an extracting agent (PND F 16.1: 2.2.22-98, 1998).

For evaluation of the ecological condition, the soil phytotoxicity (germination ability, length of roots and spouts), carbon dioxide emission, the activity of soil enzymes (catalase and dehydrogenases) were determined. The condition of soil without soil, but with the application of similar amounts of bioremediation agents to the petroleum hydrocarbon contaminated Haplic Chernozem Calcic was evaluated to determine the toxic level of the proper bioremediation agents.

Soil phytotoxicity was assessed using standard procedures to evaluate the plant shoot and root lengths. The phytotesting of contaminated Haplic Chernozem Calcic was performed using the garden radish (*Raphanus sativus* var. radicula Pers) cultivar "Rubin".

When increasing the exposition term due to petroleum hydrocarbon degradation  $CO_2$  content increases in soil air. When adding bioremediation agents, the degradation rate increases. The intensity of soil degradation was evaluated by means of gas analyzer TESTO-535 with error of ±50 ppm.  $CO_2$  emission in mg/kg for soil was recalculated by Equation (Kadulin et al., 2017):

$$C = \frac{Dc}{dt} \times \frac{V}{S} \times 1000 \times 60$$
<sup>(1)</sup>

where Dc - carbon dioxide emission, ppm

dt - time period, within which the measurement was performeds

- V chamber volume, m<sup>3</sup>
- S chamber cross-section area, m<sup>2</sup>

The activity of enzymes of oxidoreductase class (catalase, dehydrogenases) was studied by per A.Sh. Galstyan, F.Kh. Khaziyev (Kazeev et al., 2016). The enzyme activity was studied as per the recommendations of Galstyan (1978) at natural pH of the soil.

The catalase activity ( $H_2O_2$ :  $H_2O_2$  – oxidoreductase, EC 1.11. 1.6) was determined by the gasometric method proposed by Galstyan (1978) considers the quantity of decomposed peroxide during reaction with soil by volume of extracted oxygen displacing water from the burette. The enzyme activity was expressed in ml  $O_2$  g<sup>-1</sup> min<sup>-1</sup>.

The activity of dehydrogenases (substrate: NAD(P) – oxidoreductase, EC 1.1.1) was determined as per Galstyan (1978) on the restoration of tetrazolium chloride salts in triphenylformazan. The enzyme activity was expressed in mg triphenylformazan 10 g<sup>-1</sup> 24 h<sup>-1</sup>.

The number of soil bacteria was determined by the luminescence microscopy method considering the number of soil bacteria after staining with acridine orange (Kazeev et al., 2016). The results were expressed in 10<sup>9</sup> bacteria in 1 g<sup>-1</sup> soil (Equation 2).

$$M = \frac{4 \times A \times H \times 10^{10}}{p}$$
(2)

where M - number of cells per 1 g of soil

- A the average number of cells within one field of vision
- H dilution index
- P the area of the field of vision,  $\mu m^2$

#### Statistical analyses

Statistical processing of the data obtained was carried out using the software package of STATISTICA 12.0. Statistics (mean values, dispersion) were determined, and the reliability of different samples was established by using dispersion analysis (Student t-test).

#### Results

# The residual petroleum hydrocarbon content in haplic chernozem calcic after application of the biochar

The efficiency of bioremediation agent application and change in the Haplic Chernozem Calcic ecological condition was evaluated based on petroleum hydrocarbon content in the Haplic Chernozem Calcic remaining after incubation (Figure 2).



Figure 2. Change in the petroleum hydrocarbon content on the 30th day of the experiment after the introduction of biochar (10 and 20% of the soil mass (w/w)) into the petroleum carbohydrates-contaminated Haplic Chernozem Calcic, % of the PHC-contaminated soil: A) Proportion of decomposition of petroleum hydrocarbons after biochar application; B) Change in oil content, % of the original. Note: BC, biochar; PHC, petroleum hydrocarbon

Petroleum hydrocarbon content after incubation with bioremediation agents was changed to a variable degree. Maximum decrease of petroleum hydrocarbon content by 17% lower than in the petroleum hydrocarbon contaminated variant has been detected in the sample with biochar in the amount of 10% from soil mass. Such an effect is conditioned by the physical structure of the bioremediation agent. By composition, porosity and area of surface biochar is similar to the activated carbon, but it has a wider range of feedstock (Doumer et al., 2016). Within the last years biochar is actively used as an organic fertilizer during restoration of the soil fertility and agricultural functions. In the case of petroleum hydrocarbon and petroleum hydrocarbon product contamination, biochar application has an effect on microbiological activity stimulation. The preparation based on brown coal "Gumikom" is sufficiently efficient during bioremediation of petroleum hydrocarbon contaminated soils (Yagafarova et al., 2016).

# Change in the number of soil bacteria of non-contaminated and petroleum hydrocarbon contaminated chernozem after application of the biochar

The number of soil bacteria in pure soil without petroleum hydrocarbon after biochar application in the amount of 20% from soil mass (w/w) has increased by 243% from the control accordingly (Figure 3). The final calculation of the number of soil bacteria was performed using Equation 2. When applying biochar in the amount of 10% from soil mass, the number has increased only by 15%.

Upon applying biochar in amount of 10 and 20% from soil mass (w/w), the number of soil bacteria was 209 and 203% (143 and 138% from petroleum hydrocarbon contamination) from the control.





# Change in carbon dioxide emission of uncontaminated and contaminated haplic chernozem calcic after application of the biochar

As a result of natural transformation and degradation process, petroleum hydrocarbon decomposes in the soil very slowly. When adding bioremediation agents, the rate of petroleum hydrocarbon degradation increases and causes the formation of carbon dioxide and water vapours. The biochemical condition of the soils is evaluated not only by the activity of soil enzymes and microbiological indices but also by the products characterizing petroleum hydrocarbon decomposition (carbon dioxide and water vapours). In soils, the carbon dioxide emission is an index, which allows evaluating its air conditions, as well as providing indirect presentation about its microbiological activity (Galstyan, 1961).



Figure 4. Change soil respiration (CO<sub>2</sub> emission) when adding biochar (10 and 20% of the soil mass (w/w)) to petroleum hydrocarbon-contaminated Haplic Chernozem Calcic in comparison with clean soil, % of control Note: BC - biochar; PHC, petroleum hydrocarbon

petroleum For correct evaluation of hydrocarbon degradation to simple decomposition products (carbon dioxide and vapors), uncontaminated water and petroleum hydrocarbon contaminated soil samples were analyzed (Figure 4). The final calculation was performed by Equation 1. After the 1st day of the experiment,  $CO_2$ emission in the petroleum hydrocarbon contaminated Haplic Chernozem Calcic and when adding biochar didn't significant. On the 18th day, the growth of  $CO_2$  emissions was observed in the variants with biochar in amount of 10% from soil mass by 70-85% in relation to petroleum hydrocarbon contamination and then decreased to soil with biochar application in amount of 20% from soil mass – 222-362 ppm. In amount of 20% from soil mass, biochar didn't have a stimulating influence on biota and didn't cause inhibition of the biota activity compared with petroleum hydrocarbon contamination.
# Change in the catalase activity of uncontaminated and contaminated haplic chernozem calcic after application of the biochar

The enzyme activity of soils was evaluated by the catalase and dehydrogenase activity (class oxidoreductases). Enzymes of class oxidoreductases shall be informatively used in soil contamination of different chemical substances in the south of Russia (Kolesnikov et al., 2011, 2012; Minnikova et al., 2019, 2020).





Change in the catalase activity in the pure soil without petroleum hydrocarbon contamination when applying a large amount of biochar (20%) from soil mass (w/w)) was inhibited by 15% (Figure 5). With an increase in the concentration of biochar up to 20% from soil mass (w/w), the activity of catalase does not change in comparison with oil-contaminated soil. When petroleum hydrocarbon applying the catalase activity decreased by 27%. After the introduction of biochar in the amount of 10 and 20% of the soil mass (w/w), Haplic Chernozem Calcium contaminated with petroleum hydrocarbons, the activity of catalase decreased by 22 and 33% of control, respectively. After biochar was added 10% of the soil mass (w/w), an increase in activity by 10% was observed.

# Change in the dehydrogenases activity of uncontaminated and contaminated haplic chernozem calcic after application of biochar

The dehydrogenases activity changed on different sides (Figure 6). In non-contaminated soil, at the amount of 20% from soil mass stimulation by 10% in relation to the control was observed. In the petroleum hydrocarbon contaminated Haplic Chernozem Calcic, the dehydrogenase activity was higher when applying biochar in the amount of 10%from soil mass by 49% from the control (30% from petroleum hydrocarbon contamination). Petroleum hydrocarbon application to the soil increased the dehydrogenases activity conditioned oxidationreduction processes in the contaminated soil using hydrogen and carbon ions (Lopes et al., 2021). Enzymes of the oxidation-reduction group are associated with carbon and nitrogen and faster response to the application of carbon-bearing compounds than enzymes involved in the sulphur and phosphorus cycle.



Figure 6. Changes in the activity of dehydrogenases upon the introduction of biochar (10 and 20% of the soil mass (w/w)) into petroleum hydrocarbon -contaminated

Haplic Chernozem Calcic compared to control soil, mg TPF 10 g soil  $^{\rm 12}$  24  $h^{\rm -1}$ 



# Change in the intensity of early growth and development of radish seeds in uncontaminated and contaminated haplic chernozem calcic after application of the biochar

Taking into account the results of change in the enzyme activity and  $CO_2$  emission the influence on the plants as sensitive test objects were considered. For evaluation of soil toxic level after bioremediation agent application the sensitive phytotest (radish seeds) was used. Radish use allows fast response to petroleum hydrocarbon contaminated Haplic Chernozem Calcic toxic level change within a short time, particularly, when using nonorganic bioremediation agents (Minnikova et al., 2019, 2020). The toxic level was evaluated based on the germinating ability of radish seeds and its morphological indices: length of shoots and roots (Figure 7). In non-contaminated soil germinating ability of radish seeds didn't change after biochar adding. In the petroleum hydrocarbon contaminated soil germinating ability decreased by 45%. When adding biochar in 10 and 20% from soil mass, germinating ability increased by 17 and 44% from the control accordingly (by 32 and 65% in relation to the petroleum hydrocarbon contaminated soil).



Figure 7. Changes in the intensity of initial growth (length of shoots (A) and roots (B)) and indicators of seed germination (germination (C), view garden radish Garden radish (Raphanus sativus var. radicula Pers) cultivar "Rubin" (D)) after addition of the biochar (10 and 20% of the soil mass (w/w)) to petroleum hydrocarbon-contaminated Haplic Chernozem Calcic in comparison with uncontaminated soil.

Note: BC, biochar; PHC, petroleum hydrocarbon

The indices of early radish growth intensity after cultivation in the uncontaminated and petroleum hydrocarbon contaminated soil are shown in Figure 6. In pure petroleum hydrocarbon uncontaminated soil, the length of sprouts with biochar in the amount of 10 and 20% from soil mass was higher than the control by 53 and 55%. When adding petroleum hydrocarbon in the soil, a decrease of growth sprout by 67% in relation to the control was observed. When adding biochar in petroleum hydrocarbon contaminated soil, growth in the length of sprouts by 51 and 66% was observed for biochar in amount of 10 and 20% from soil mass accordingly.

The length of radish roots has changed based on a similar scenario. In uncontaminated soil, stimulation of the root length is detected upon biochar application in amount of 10 and 20% from soil mass by 159 and 138% higher, than the control. In oil contaminated soil without biochar, the root length is lower by 18%. Applying biochar in amount of 10 and 20% from soil mass is higher by 17 and 44% from the control (by 35 and 63% from the petroleum hydrocarbon contaminated variant).

Biochar in the amount of 20% from soil mass to be by 24% higher than for biochar in amount of 10% from soil mass has a more favourable influence on the intensity of radish seed sprouting in case of petroleum hydrocarbon contamination. Stimulation of radish root and sprout growth in the soil with biochar is conditioned with a high concentration of organic matter and mineral elements.

### Discussion

Restoration of the biological properties of the soil after petroleum hydrocarbon contamination is a long process (Figure 8). The restoration degree is evaluated based on stimulation of the soil biological indices: the

intensity of early growth and development of radish seeds, carbon dioxide emission, number of soil bacteria (R=-0.61), change in activity of catalase (R=-0.79) and dehydrogenases (R=-0.92) versus to the control. The CO<sub>2</sub> emission is closely correlated with the catalase activity – R=0.75 and inverse correlation – with the length of radish sprouts and roots – R=-0.83 and 0.94. The immobilized bacterium on the biochar treatment was maintained at the highest level during the entire remediation compared with other ameliorants (Zhang et al., 2019). At 50 days, the FDA hydrolysis, dehydrogenases and polyphenoloxidases activity in the immobilized bacteria on biochar treatment (BIM) reached maximum values and were 22.71 mg g<sup>-1</sup> 24h<sup>-1</sup>, 93.44 µg g<sup>-1</sup> 24h<sup>-1</sup> and 39.96 mg g<sup>-1</sup> 24h<sup>-1</sup>, respectively, confirming that biochar not only can provide a favorable habitat but also sufficient nutrients for microbes to produce enzymes and biodegrade the contaminants. The enzymatic activities (as catalase) decreased the possible cause of such change may be due to the partial decomposition of biochar and accumulation of residues of refractory components inhibiting the activity of microorganisms, leading to microbial exfoliation and even death.



Figure 8. Correlation dependence between the petroleum hydrocarbon content and changes in the biological parameters of the Haplic Chernozem Calcic after the introduction of the biochar

The number of soil bacteria is associated with the length of shoots and roots by a direct relationship R = 0.97 and the length of roots R = 0.89. In order to develop a new method for remediation of petroleum hydrocarbon contaminated soils, salt tolerant *Corynebacterium* variable HRJ4 and biochar were used early (Zhang et al., 2016). Results indicate that the bacteria involving the biochar were very efficient in degradation of PAHs (PYR, NAP) mixture and n-alkanes (C<sub>16</sub>, C<sub>18</sub>, C<sub>19</sub>, C<sub>26</sub>, C<sub>28</sub>).

Thus, in the case of Haplic Chernozem Calcic petroleum hydrocarbon contamination, the use of biochar in 10% is the most efficient, as it is environmentally reasonable. Higher concentration increases by germination ability and length of plant roots and sprouts and decrease of petroleum hydrocarbon content in the soil. Decomposition of low petroleum hydrocarbon concentration, 5% from soil mass, was evaluated in the research. These data are confirmed by Mukome work with coauthors when studying 0.25% petroleum hydrocarbon contamination with biochar application on the basis of pine saw dusts and straw (Mukome et al., 2020).

This research has demonstrated that biochar on the basis of pine saw dusts in the soil is efficient together with fertilizer even at low concentration of light crude petroleum hydrocarbon in the soil. However, when using biochar from wheat straw, the significant difference between the control soil with crude petroleum hydrocarbon and treated by biochar applying in amount of 1% from petroleum hydrocarbon amount in the soil (Han et al., 2016).

Biochar has displayed a good potential to remediation of petroleum hydrocarbons owed to its wide availability of the necessary feedstock, sustainable nature, high efficiency, large internal surface area and desirable physicochemical surface properties (Zahed et al., 2021). Using biochar can include carbon sequestration, improving soil fertility, reclamation, and processing of agricultural wasting.

# Conclusion

Biochar use contributes to accelerating petroleum hydrocarbon degradation and improving Haplic Chernozem Calcic ecological condition after petroleum hydrocarbon contamination. Use of biochar in the amount of 10 % from Haplic Chernozem Calcic mass (w/w) is more reasonable than biochar amount of 20% mass (w/w). Petroleum hydrocarbon degradation intensity and stimulation of most of Haplic Chernozem Calcic biological activity indices were higher when using 10% from biochar concentration. A decrease in the

concentration of petroleum hydrocarbons in the soil after the introduction of biochar increases the activity of catalase (R = -0.79), dehydrogenases (R = -0.92) and the number of soil bacteria (R = -0.61). The use of biochar accelerates the decomposition of petroleum hydrocarbons and improves the ecological state of the soil after contamination with petroleum hydrocarbons.

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# Effects of low-intensity fire on soil organic carbon stocks and physicochemical properties in the Mediterranean ecosystem Mehmet Parlak \*

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# Abstract

Due to inherent climate characteristics, forest fires are commonly encountered in the Mediterranean ecosystem. Forest fires influence water resources, flora, fauna, air quality and soil properties. This study was conducted to determine the effects of a low-intensity fire in Lapseki - Dışbudak village on soil physical and chemical properties. Soil samples were taken from 0-5 cm depth on unburned (control) and burned lands 1 month and 3 years after the fire and samples were analyzed for organic carbon stock, texture, aggregate stability, bulk density, pH, electrical conductivity (EC), lime, organic matter, organic carbon, exchangeable K, Ca, Mg, Na, and available Fe, Cu, Mn, and Zn.While the average pH, EC (dS m<sup>-1</sup>), exchangeable Ca (mg kg<sup>-1</sup>), Na (mg kg<sup>-1</sup>), available Mn (mg kg<sup>-1</sup>) and Zn (mg kg<sup>-1</sup>) values were respectively measured as 6.37, 0.72, 3504.10, 34.97, 202.31 and 4.23 in burned lands in the 1st month after fire, the values were respectively measured as 6.25, 0.53, 2870.90, 24.89, 127.96 and 2.71 in control areas. At the end of the 3rd year, available Mn was measured as 81.69 mg kg<sup>-1</sup> in burned lands and 53.58 mg kg<sup>-1</sup> in unburned lands. It was concluded that at the end of 3 years, lowintensity fire was effective in improving soil properties.

**Keywords**: Low intensity fire, soil organic carbon stock, soil properties, soil nutrient.

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# Introduction

Forest fires constitute a serious problem especially in the Mediterranean climate zone with hot and dry summers (Alcañiz et al., 2016). The fires encountered in the Mediterranean ecosystem recently increased as related to land use change and climate change (Fernández-García et al., 2019). The Mediterranean climate domain extends to a much larger geographical scale globally including areas as far away as the southern parts of California, and some regions in Australia, New Zealand, and Argentina (Zdruli et al., 2011). There is some research on the effects of fires in the Mediterranean ecosystem on soil properties (Parlak, 2018; Hueso-Gonzalez et al., 2018; Fernandez- Garcia et al., 2019; Merino et al., 2019; Moya et al., 2021; Dindaroglu et al., 2021). Impacts of fires vary with the vegetation, topography, soil properties, fire severity and post-fire use conditions (Certini, 2005).

Soil organic carbon is a highly significant indicator in terms of land degradation neutrality, sustainable use of soils and mitigation of the negative impacts of climate change (Knicker, 2007; Yigini and Panagos, 2016). Organic carbon stocks in forest soils are largely influenced by fires (Lal, 2005). Vega et al. (2013) indicated that low intensity fire in *Pinus pinaster* stands did not increase soil organic carbon, available P and K levels. Stinca et al. (2020) indicated that fires in beech forests of southern Italy did not generate a change in organic C stocks.

Although research was conducted in Turkey about the effects of low intensity fires on soil physical and chemical properties (Ekinci, 2006; Kara and Bolat, 2009; Akburak et al., 2018; Camci Çetin et al., 2019;

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Publisher : Federation of Eurasian Soil Science Societies e-ISSN : 2147-4249 Dindaroğlu and Turan 2019), there are no studies encountered in the literature about the effects of low intensity fires on soil available micro element contents (Fe, Cu, Mn, Zn). This study was conducted to determine the effects of low intensity forest fires encountered in the Mediterranean ecosystem on soil organic carbon stocks and some physicochemical properties (texture, aggregate stability, bulk density, pH, electrical conductivity, lime, organic matter, organic carbon, available P, K, exchangeable Ca, Mg, Na, available Fe, Cu, Mn, and Zn).

# **Material and Methods**

### Study area

Lapseki town in Çanakkale province is located in the southern Marmara section of Marmara region in the northwest of Biga peninsula. Lapseki is geographically positioned between 40° 20' north latitude and 26° 42' east longitude. Lapseki is surrounded by the Marmara Sea to the north, central town of Çanakkale to the south, the Dardanelles to the west and Biga town to the east (Figure 1). Lapseki has a semi-humid Marmara climate (a transitional climate between Mediterranean and Black Sea climates) (Koçman, 1993). According to long-term averages (1929 – 2020), total annual precipitation in Lapseki is 624 mm and the average temperature is 15.1 °C (MGM, 2021). The slope in Dışbudak forest lands is about 9% and the forest mostly comprises red pine (*Pinus brutia* Ten.), kermes oak (*Quercus coccifera*), cretan rock rose (*Cistus creticus* L.), mock privet (*Phillyrea latifolia* L) and summer asphodel (*Asphodelus aestivus* Brot.). According to the World Reference Base for Soil Resources, Dışbudak forest soils are classified as Lithic Leptosol (WRB, 2015). In an anthropogenic fire case encountered in Lapseki-Dışbudak village in September 2006, about 5 ha forest land was burned, the organic layer was over-burned, but sub soil was not influenced.



Figure 1. Location of study area

### Soil sampling

Attempts were made to consider similar slope, aspect and elevation while taking samples from Lapseki-Disbudak forest lands. In each sampling period (1 month and 3 years after the forest fire), 9 disturbed and undisturbed soil samples were taken from unburned (control) lands and 9 samples from burned lands and coordinates of the sampling points were recorded with a GPS (Global Positioning System) device. Samplings were made in accordance with the random sampling system. Following the careful removal of litter, soil samples were taken from 0 – 5 cm soil depth with a stainless-steel shovel and steel sampling cylinders. Soil samples were taken in the 1<sup>st</sup> month and 3<sup>rd</sup> year following the fire. A total of 36 disturbed and undisturbed soil samples were taken with 18 from unburned lands (9 samples x 2 periods x 1 depth) and 18 from burned lands. Soil samples were brought to the laboratory, air dried and passed through a 2 mm sieve.



Figure 2. Control lands (A) and burned lands (B) 1 month after the forest fire

### Soil analysis

For texture analysis, 50 g soil sample was kept in 10% calgon solution overnight, then placed in 1130 ml glass cylinders and a hydrometer was used to examine sand, silt and clay fractions (Gee and Or, 2002). Aggregate stability, an indicator of changes in soil structure, was determined with micro aggregates less than 0.25 mm in diameter (Nimmo and Perkins, 2002). Soil bulk density was determined in undisturbed soil samples by drying samples in an oven at 105 °C (Grossman and Reinsch, 2002). The pH of the samples was determined in saturated paste extract with a pH meter (Thomas, 1996) and electrical conductivity (EC) of the same extract was determined with an EC meter (Rhoades, 1996). Lime content was determined with a Scheibler calcimeter by measuring the CO<sub>2</sub> gas released when soil was contacted with HCl (Loeppert and Suarez, 1996). Organic matter and organic carbon were determined with the modified acid digestion method (Nelson and Sommers, 1996). For soils with a pH of less than 7, available phosphorus (P) was determined with the use of Bray and Kurtz's (1945) method; exchangeable potassium (K), calcium (Ca), magnesium (Mg) and sodium (Na) were determined in 1 N ammonium acetate (NH<sub>4</sub>OAc) extracts from the samples. Extract K and Na contents were determined with the use a flame photometer (Helmke and Sparks, 1996) and available Ca and Mg contents were determined with an ICP-OES ((Inductively Coupled Plasma - Optical Emission Spectrometry) (Suarez, 1996). Available Fe, Cu, Zn, Mn and Zn contents were determined in 0.005 M diethyl triamine penta acetic acid (DTPA), 0.01 M calcium chloride (CaCl<sub>2</sub>) and 0.1 M tri ethanol amine (TEA) mixture solution (pH: 7.3) extracts with the use of an ICP-OES device (Lindsay and Norvell, 1978).

Soil organic carbon stock (SOCS) was determined with the use of the soil depth equation (Gross and Harrison, 2018).

where, BD is bulk density (g cm<sup>-3</sup>); D is sampling depth (m); and TOC is soil organic carbon content (g kg<sup>-1</sup>).

### Statistical analysis

For comparison of soil properties in control (unburned) and burned lands, the paired t-test was used for data exhibiting normal distribution and Mann-Whitney U-test was used for data without normal distribution. The Levene test was used to check variance homogeneity and Shapiro-Wilk test was used to check normality pre-conditions. Statistical analyses were conducted with the use of SPSS Statistical Package v.20.0 (IBM, 2011). Significant means were compared with the use of Duncan's test at p<0.05 significance level.

### **Results and Discussion**

In the 1st month samples, differences in soil pH, EC, available Ca, Na, Mn and Zn contents of the control and burned lands were found to be significant at p<0.05 level, but the differences in SOCS and the other soil properties (sand, silt, clay, aggregate stability, bulk density, lime, organic matter, soil organic carbon, available P, exchangeable K, Mg, Fe, Cu, and Zn) were not found to be significant (Table 1). Increasing pH and EC levels of soil samples taken 1 month after the fire were attributed to soluble salts released during combustion of organic matter (Pereira et al., 2014; Muñoz-Rojas et al., 2016; Parlak, 2018). Soil temperature could not be measured during the forest fire. Effects of fire on pH largely relies on soil temperature during the fire event. Parlak (2011) indicated decreasing soil pH levels at 200 °C, but increasing pH levels at 500 °C. Merino et al. (2019) conducted a study in three study sites in central Spain (*Pinus nigra* forest, *Pinus pinaster* forest, Cytisus oromediterraneus shrubland) and indicated that prescribed fires did not alter the soil organic matter content. Kutiel and Inbar (1993) took soil samples from 0-5 cm depth around burned pine trees and reported increased exchangeable Ca and Na contents. Pardini et al. (2004) indicated that oak forest fire increased exchangeable Ca, but decreased exchangeable K content of the samples taken from 0-10 cm depth. Within 1 month of sampling following the fire, precipitation resulted in soil erosion in the study area. Caon et al. (2014) indicated that soil erosion resulted in loss of ash accumulated on the soil surface after the fire. Exchangeable cations (especially Ca<sup>2+</sup> and Mg<sup>2+</sup>) are not lost through volatilization, but lost through leaching and runoff (Parlak, 2018). Among the exchangeable cations, Ca<sup>2+</sup> is influenced most by the fire. Lasanta and Cerdà (2005) reported high Ca concentration in runoff 7 months after the fire. Scharenbronch et al. (2012) reported that low-severity prescribed fire in USA oak forest increased extractable Na. Akburak et al. (2018) indicated that low-intensity prescribed fire slightly increased soil pH, but did not increase EC, organic carbon, available K and Mg contents. Kaptanoğlu et al. (2018) conducted a study in mixed broad-leaved and coniferous forest in northwest Turkey, Bursa-Uludağ, and indicated that surface fire increased soil pH and Ca. Camci Cetin et al. (2019) reported low intensity forest fire in scots pine and black pine stands only increase the electrical conductivity among soil chemical parameters.

Relations of Fe, Cu, Mn and Zn-like micro elements with the fire are not known due to lack of specific studies (Certini, 2005). Gonzales Parra et al. (1996) indicated that total and available manganese contents significantly increased after fires. With fire-generated ash, Mn is released into soil in amorphous and crystalline oxide forms. Probably Fe, Cu and Zn also exhibit similar behavior with Mn and move slightly downward (Certini, 2005). García-Marco and González-Prieto (2008) indicated that controlled fire resulted in changes in the availability of micro elements in the short term, increased available Mn and Zn and did not influence Cu availability. Gómez-Rey et al. (2013) indicated that controlled fire in NW Spain did not change soil extractable Fe levels significantly. Norouzi and Ramezanpour (2013) indicated that forest fires did not have significant effects on soil available Cu content. Mitic et al. (2015) investigated the effects of forest fires in Vidlic mountain (Serbia) on soil Zn and Cu content and reported that fires influenced soil Zn content, but did not influence Cu content. Availability of soil nutrients is generally related to ash generated by the fire. Other researchers (Neill et al., 2007; Switzer et al., 2012; Roaldson et al., 2014; Chen et al., 2019) indicated that low-intensity fires did not influence soil carbon pools. Alcañiz et al. (2018) indicated that soil texture, bulk density, and soil aggregate stability did not change following a low-intensity fire. Alterations of soil physical properties by a fire depend on fire intensity, severity and recurrence (Parlak, 2018). Since soil temperature is not high in low-intensity fires, soil physical properties do not change.

Table 1. Soil organic carbon stocks and some physicochemical properties of soil samples were examined 1 month after the forest fire (mean ± standard deviation)\*

Soil proportios	Control area			Durr	Purned area			Mann- Whitney
Son properties	COI	uoi	alea	DUIT	burneu area		t-test (p)	U test (p)
SOCS (ton ha <sup>-1</sup> )	21.69	±	5.61	25.07	±	1.33		0.1120
Sand (%)	48.83	±	4.67	47.25	±	6.08		1.0000
Silt (%)	24.76	±	2.19	25.75	±	2.46		0.6588
Clay (%)	26.85	±	4.93	27.00	±	2.71		0.6272
Aggregate stability (%)	37.97	±	9.76	36.08	±	7.44		0.6588
Bulk density (g cm <sup>3</sup> )	1.45	±	0.04	1.43	±	0.04		0.5660
рН	6.25	±	0.13 b	6.37	±	0.08 a		0.0273*
EC(dS m <sup>-1</sup> )	0.53	±	0.07 b	0.72	±	0.05 a		0.0009*
CaCO <sub>3</sub> (%)	0.67	±	0.37	0.58	±	0.21	0.7240	
Organic matter (%)	5.16	±	1.35	6.03	±	0.28		0.0521
Organic carbon (g kg <sup>-1</sup> )	29.98	±	7.87	34.99	±	1.65		0.1853
Available P (mg kg <sup>-1</sup> )	17.22	±	6.24	18.77	±	7.64		0.8253
Exchangeable K (mg kg <sup>-1</sup> )	266.11	±	94.23	298.56	±	88.18		0.4268
Exchangeable Ca (mg kg <sup>-1</sup> )	2870.90	±	384.50 b	3504.10	±	210.30a		0.0006*
Exchangeable Mg (mg kg <sup>-1</sup> )	400.70	±	100.90	517.30	±	103.80		0.0521
Exchangeble Na (mg kg 1)	24.89	±	4.81 b	34.97	±	5.96 a		0.0036*
Available Fe (mg kg <sup>-1</sup> )	4.69	±	0.95	4.67	±	0.90		0.7510
Available Cu (mg kg <sup>-1</sup> )	1.47	±	0.48	1.35	±	0.14		0.7911
Available Mn (mg kg <sup>-1</sup> )	127.96	±	25.13 b	202.31	±	37.16 a		0.0020*
Available Zn (mg kg-1)	2.71	±	0.84 b	4.23	±	1.03 a		0.0081*

\* The means indicated with different letters are significantly different at 5% level.

In soil samples taken in the 3rd year, only differences in Mn contents of the control and burned lands were found to be significant, but the differences in SOCS and the other soil properties were not found to be significant (Table 2). Fonseca et al. (2017) worked on a low severity fire in Montesinho Natural Park of northeast Portugal and indicated that soil organic matter, pH and EC values returned to pre-fire levels 36 months after the fire. Moya et al. (2019) took soil samples 3 years after a low-medium severity fire in *Pinus halepensis* Mill forest in the Mediterranean ecosystem and did not encounter significant changes in soil texture, organic carbon and pH. After the low-intensity fire in Lapseki-Dişbudak forest, 3 years were sufficient for the improvement of soil physicochemical properties. Low severity fires may have positive or negative effects on the environment, but previous studies mostly reported improved environments (Alcañiz et al., 2018). Low-severity fires may aid in replenishment of ecosystems and reduce the risk of forest fires.

Table 2. Soil organic carbon stocks and some physicochemical properties of soil samples were examined 3 years after the forest fire (mean±standard deviation)\*

Soil proportion	Control area			Dum	Burned area			Mann- Whitney
Son properties	COIL	1101	area	DUIT	Burned area		t-test (p)	U test (p)
SOCS (ton ha <sup>-1</sup> )	16.03	±	4.88	19.11	±	2.72		0.2164
Sand (%)	47.09	±	3.06	45.99	±	3.17		0.2893
Silt (%)	26.39	±	3.62	26.83	±	2.58		0.5660
Clay (%)	26.50	±	4.02	27.17	±	2.38		0.9648
Aggregate stability (%)	46.28	±	5.77	45.13	±	10.29		0.8598
Bulk density (g cm <sup>3</sup> )	1.44	±	0.05	1.45	±	0.05		0.5660
рН	6.48	±	0,20	6.80	±	0,48		0.1223
EC(dS m <sup>-1</sup> )	0.42	±	0,10	0.45	±	0,12		0.6911
CaCO <sub>3</sub> (%)	0.85	±	0.22	0.79	±	0.11		0.2510
Organic matter (%)	3.29	±	0.33	3.02	±	0.52		0.0703
Organic carbon (g kg <sup>-1</sup> )	22.40	±	7.09	26.33	±	3.63		0.1853
Available P (mg kg <sup>-1</sup> )	14.11	±	2.61	16.00	±	3.08		0.2004
Exchangeable K (mg kg <sup>-1</sup> )	251.67	±	64.05	261.44	±	37.00		0.6588
Exchangeable Ca (mg kg <sup>-1</sup> )	2533.00	±	391.00	2628.00	±	416.80		0.7239
Exchangeable Mg (mg kg <sup>-1</sup> )	341.65	±	37.33	349.87	±	28.56		0.6588
Exchangeble Na (mg kg <sup>1</sup> )	24.04	±	3.85	22.93	±	4.07		0.6588
Available Fe (mg kg <sup>-1</sup> )	1.67	±	0.19	1.98	±	0.43		0.0637
Available Cu (mg kg <sup>-1</sup> )	0.87	±	0.21	0.83	±	0.14		0.8253
Available Mn (mg kg <sup>-1</sup> )	53.58	±	16.00 b	81.69	±	10.37 a		0.0027*
Available Zn (mg kg <sup>-1</sup> )	1.75	±	0.37	1.98	±	0.45		0.2893

\* The means indicated with different letters are significantly different at 5% level.

# Conclusion

This study was conducted to investigate the effects of a low-severity fire in the Mediterranean ecosystem on soil properties. At the end of the 3rd year after the fire, available Mn increased in burned lands, but the other soil properties (organic carbon stock, texture, agregate stability, bulk density, pH, electrical conductivity, lime, organic matter, organic carbon, exchangeable K, Ca, Mg, Na, available Fe, Cu, Mn, and Zn) did not change. Further research is recommended about low-intensity fires in different ecosystems including lands with a high risk of desertification. To prevent large fires with devastating impacts on the ecosystem, low-intensity fires could be recommended.

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# Effect of climatic conditions on the productive and biochemical characteristics of grape varieties grown on sierozem soil

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# Abstract

The aim of this study was to evaluate the effect of climatic conditions on phenological observations, yield components and biochemical characteristics of grape varieties grown on sierozem soil of Turkestan region, Southern Kazakhstan. A total of 13 different medium and late grape ripening varieties known, loved and widely used by the local people for many years in cultivation were chosen. The phenological observations (date of budding and removable maturity date of grape) yield components (yield of grape, weight of bunch of grape, number of bunch of grapes, as well as productivity) and the biochemical characteristics (titratable acidity, glucoacidimetric index, glucose content, fructose content) of grape varieties were evaluated in three seasons (2018-2020). During the vegetation period, date of budding and removable maturity date observations were made and recorded as day/month. As a result of the study, differences were found among local genotypes in terms of phenological stages. On the average of years, the earliest date of budding was recorded on 21 March with Children's early and the latest date of budding was observed on 02 April Moldova and Victory. The highest yield and productivity were obtained from varieties Husayn kelin barmak and Chocolate grape varieties. Titratable acidity range was between 5 g/L – 8g/L, in average of years, the highest titratable acidity in late ripening grape varieties was found in Victory (7.3 g/L) while Moldova (6.0 g/L) had the lowest. Titratable acidity of Husayn kelin barmak (7.3 g/L) was determined also higher than the medium ripening grape varieties while Guzal Kara had the lowest, only 5.3 g/L. Glucose and fructose contents were determined as 8.58% and 11.04 in different grape varieties, respectively.

Keywords: Sierozem soil, grape, climate, sugar, phenological observations.

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# Introduction

Grapes are one of the world's most commonly produced fruit crops. In addition to the increasing consumption of fresh grapes, in recent years, interest in products made from grapes has greatly increased, particularly in grape juices (FAO-OIV Focus, 2016). The numerous health benefits may be part of the reason because these products are rich sources of phenolic compounds (phytochemicals with potential anti-oxidant activity) (Granato et al., 2016). A brief characterization of the global viticulture and winemaking sector is provided upfront to better understand its relevance in the world economy. As detailed in the report of the FAO-OIV Focus, (2016), it is estimated that the world vineyards cover an area of approximately 7.449 million ha (2018). (Santos et al., 2020)

Although the suitability of a region for grapevine cultivation is largely controlled by growing season mean temperature that should range between 12 and 22°C, other atmosphere-driven conditions, such as growing

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Publisher : Federation of Eurasian Soil Science Societies e-ISSN : 2147-4249 season length, radiation levels, winter minimum temperatures, spring and fall frosts or soil water balance, among others, are also important limiting factors (Jones et al., 2012). Air temperature plays a central role in determining grapevine phenology (Bonada et al., 2015), influencing the onset of phenological stages like budbreak, flowering and veraison and the phenophase intervals (Fraga et al., 2016). Temperature also affects grapevine yield (Bock et al., 2010), wine production (Santos et al., 2013) and quality (de Orduña, 2010). Relatively constant and moderate temperatures during ripening favour the biochemical processes of colour, flavour and aroma development in grape berries. For rainfed grapevines, the water balance is mainly determined by precipitation, atmospheric humidity and soil water holding capacity. The amount of annual precipitation and its seasonal distribution are also crucial to the evolution of the plant water status, with subsequent effects on berry quality. Moreover, under extremely dry atmospheric conditions, stomata can close to preserve moisture, ceasing photosynthesis (Ashenfelter and Storchmann, 2014). Nevertheless, some reported effects of water stress on grape berry quality attributes are contradictory, being strongly dependent on the local conditions, on the degree of water stress and on the period in which it occurred (Costa et al., 2020)

In Southern Kazakhstan fruit and grapes are cultivated with seed-fruits covering 70% of the planting area. Stone-fruits occupy 20%, and nut plantations and berries take up to 10%. In the South-eastern zone of Kazakhstan, with an abundance of solar heat and the availability of irrigation water, the vineyards make it possible to get a good harvest and can be highly profitable. With a high potential opportunity, the productivity of a modern vineyard in Kazakhstan remains low. It is known that the main factor for increasing the productivity of grape plantations is the assortment, which is improved both by introducing varieties based on soil-climatic analogues, and by breeding and introducing genotypes created by methods of combinational selection on a genetic basis. Today, in our country, the planting area of vineyards is about 15 000 ha, new territories are being developed, and serious research work is being done by scientific research institutes.

The aim of this study was to evaluate the effect of climatic conditions on phenological observations, productive and biochemical characteristics of grape varieties grown on sierozem soil of Turkestan region, Southern Kazakhstan.

# **Material and Methods**

### **Study sites**

The experiment was performed at Saryagash district of the Turkistan region, South Kazakhstan (Figure 1). The experimental fields had been in grape growing regions of South Kazakhstan from 2015. Grape (*Vitis vinifera*) is the fruit crop in this region. This region is characterized by a semi-arid climate. Most of the precipitation occurred in October to May.



#### Soil

The main soil type, which is typical for the region, is sierozem soils. The sierozem soils are found in arid regions which characterized by a brownish-gray surface on a lighter layer based on a carbonate or hard-pan layer (USDA, 1999). Ordinary sierozems develop on loess-like loams and have fully developed profile with a rather noticeable division into genetic horizons. Sierozems are marked by good water-physiological properties, high biological activity, and adequate fertility; they produce high yields when irrigated. There are various subtypes: light, conventional (standard), dark, and northern (Saparov, 2014; Absatova et al., 2022). The soil belongs to the general soil type of dark sierozem. The soil pH was 8.05 in 0-30 cm soil dept, 7.86 in 30-60 cm soil dept, soil organic matter was 1.50% in 0-30 cm soil dept, 0.55% in 30-60 cm soil dept, mineral-N was 54.2 mg/kg in 0-30 cm soil dept, 41.5 in 30-60 cm soil dept, available phosphorus was 10

mg/kg in 0-30 cm soil dept, 8 in 30-60 cm soil dept, and exchangeable potassium was 401 mg/kg in 0-30 cm soil dept, 228 in 30-60 cm soil dept.

#### Grape varieties (Vitis vinifera)

A total of 13 different grape varieties including 8 medium ripening varieties (Guzal Kara, Akhtamar, Globus, Children's early, July, Queen of the Vineyards, Husayn kelin barmak and Husayn red) and 5 late ripening varieties (Taifi pink, Zebo, Moldova, Pobeda and Chocolate) which is known and widely used by the local people for many years used in this study (Figure 2).



Moldova

Chocolate

Figure 2. Medium and late ripening grape varieties at Saryagash district of the Turkestan region, South Kazakhstan

Pobeda

#### **Experimental pilot**

The study was conducted in an experimental site located at Saryagash district of the Turkistan region, South Kazakhstan, during three consecutive seasons performed within three years (2018–2020). Grape plants were cultivated in 2015, using trellis culture and dragon-shaped pruning. Row spacing was 3m, plant spacing was 1.5m. During the field experiment (2018-2020), every year same agricultural practices used in same plot of field experiment. During the trials, all the cultural practices regarding fertilization, irrigation, weed control, and pest and disease management were conducted as standard regional cultivation practices.

#### Harvest and measurements

During the maturity period of these grape cultivars in the pilots, we picked 3-5 clusters from 3 plants of every grape variety, 10 grape berries were randomly collected from the head, middle and bottom of grape clusters. During the vegetation period, date of budding and removable maturity date of grape were recorded as day/month. The plots were harvested at full maturity stage of the grapes. At harvest, the number of bunches per vine and their masses were recorded to estimate the yield of grape (t ha<sup>-1</sup>), weight of bunch of grape (gr) and number of bunch of grape (pics/branch). The harvested all grape samples were placed in plastic bags with dry ice and immediately transported to our laboratory for biochemical characteristics of.

For the biochemical characteristics of the each grape varieties, titratable acidity, glucoacidimetric index and sugars (glucose and fructose) were determined. The titratable acidity of each grape cultivar was analyzed by titrating the grape juice pH to 8.2 with NaOH and was expressed as gram tartaric acid equivalents per liter of juice (AOAC, 2000). The reducing sugars were determined according to the Somogy-Nelson colorimetric method (Nelson, 1944). Glucoacidometric index was calculated as the ratio of sugars (%) and titratable acidity.

# **Results and Discussion**

### **Climate conditions**

Analyzing the weather conditions in Turkestan region of the south of Kazakhstan during the study periods, it was noted that the average annual air temperature in the long term period from 2009 to 2020 relative to the period 2018-2020 increased by 2°C. The average annual maximum and minimum temperatures also increased, and in absolute terms, the change in temperature was 2°C and 3°C, respectively. At the same time, the absolute minimum temperature decreased by 6°C, from -4 to -10 °C (Figure 3). During the period of active growth and ripening of grapes (July), the greatest changes in air temperature were noted. Thus, the average temperature increased by 3° C in 2019, and the maximum and minimum temperatures increased by 3° C. At the same time, moisture supply during the period of active growth and ripening of grape varieties decreased in July 2019, although the annual amount of precipitation increased in August 2020.

Temperature is considered a fundamental driver of plant development and phenological cycles. It can modify profoundly the timing of cycles and such changes have been documented in many studies for different plant species under increasing temperatures in the past. During the ripening period, the air temperature plays a determinant role for grape maturation, including the aroma and the coloration, having an important effect on the characteristics of the wines (Jackson and Lombard, 1993). The day temperatures influence the coloration, but the effect of conditions of cool nights temperatures on it is even stronger (Kliewer, 1973). Several studies have indicated that increased temperatures have been associated with earlier phenological development of many wild and cultivated plants (Chmielewski et al., 2004; Cleland et al., 2007; Gordo and Sanz, 2009; Menzel et al., 2006; Schwartz et al., 2006), however, delays have also been documented for specific events, notably leaf colouring and leaf fall (Menzel et al., 2003; Estrella and Menzel, 2006; Gordo and Sanz, 2009). Phenology is modified by a complex interaction of (i) the sensitivity of the species phenology to climate drivers such as temperature but also photoperiod and water stress and (ii) the phenological stage in question.

Meteorological conditions differed between harvests, especially insolation (Figure 3). In the final stage of grape maturation, rainfall was higher in the 2018 harvest. From January to March, the cumulative rainfall was 76.97 mm in 2018, 72.30 mm in 2019 and 90.00 mm in 2020. The largest difference occurred in the first 30 days of the year. In 2018, rainfall was better distributed, while in 2019 and 2020, one periods of lower water availability occurred in the March (Figure 3), which resulted in lower soil water storage. The total hours of annual insolation (uv index) in vineyards influence the photosynthetic and metabolic activities of vines and, considering the quality of grapes, insolation has direct relation with temperature. In the 2019 harvest, insolation was higher (uv index is 9 in June and July), but in the other harvests studied in 2018 and 2020, uv index was lover (8 in June 2018 and 2020, 7 in July 2018 and 2020).

### Phenological observations

According to results, some differences were found among grape varieties in terms of phenological stages (Figure 4). On the average of years, the earliest date of budding was recorded on 21 March with Children's early and the latest date of budding was observed on 02 April Moldova and Victory. A difference of about 13 days was found between grape varieties in terms of the bud burst and full bloom dates depending on the direction and elevation of the vineyard. Changes in the phenology of several grape cultivars in recent decades are particularly in connection with the change of temperature (Figure 4). The beginning of all phenological stages in grape varieties in the period (2018-2020) occurred in earlier parts of the year, compared to the historical data. One of the most important phenological stages, date of budding, has been shifted ten-twenty days in the earlier part of the year. However, cultivars differently react to climate change. The biggest change in date of budding was observed in late ripening varieties (Taifi Pink, Zebo, Moldova, Victory and Chocolate). Medium ripening varieties (Guzal Kara, Akhtamar, The globe, Chilren's early, July, Queen of the Vineyards, Husayn kelin barmak, Husayn red) were less affected by the weather conditions compared to the late ripening varieties (Figure 4). Date of budding of late ripening varieties, in contrast to medium ripening varieties in 2018 happened 7-20 days ago than in 2019. However, date of budding of late ripening varieties, on average of 2018-2020, occurred 2-3 days earlier, compared to 2020. Phenological development stages differed in the same ecology but in different vegetation years due to changes in climate data (Cangi and Bekar, 2017). Different results were obtained from previous studies carried out in different regions regarding phenological characteristics. Budburst times of cultivars were 4-20 April (Kose, 2014).





Figure 3 Climate conditions of the experimental area, Turkestan region, Southern Kazakhstan

As a result, genetic factors and ecological conditions may be the cause of the different results in the studies performed in different regions. Different varieties within a species can have marked differences in date of budding. In the context of climate change, a deeper understanding of varietal differences in date of budding for agriculture is critical to select varieties that are adapted for production under future climate conditions.



Figure 4. Phenological observations of grape varieties grown on sierozem soil (a) Date of budding (b) Removable maturity date of grape

### Yield and yield parameters

Yield of medium and late ripening grape varieties and its components have been assessed at harvest (Figure 5). In all varieties, yields for the 2018 were higher compared to 2019 and 2020 due to a greater weight and number of bunch of grape per plant. The highest yield and productivity were obtained from varieties Husayn kelin barmak (239.1 t/ha) and Chocolate (231.7 t/ha), with an average of 183.9 t/ ha, respectively (Figure 5). Under subtropical conditions, Hernandes et al. (2010) recorded higher yields for IAC 138-22 'Máximo' than the values reported in this study for the same variety, with an average of 4.28 kg/vine and 22.5 bunches per vine. The increase in yield can be explained by the greater bunch mass found by those authors (195.8 versus 134 g). However, the productivity of 'Isabel Precoce' in the present study was higher than that found for the variety in temperate climate conditions, which produced 6.71 t/ha (Botelho et al., 2011).

The Akhtamar (medium and ripening grape variety) and Moldova (late ripening grape variety) varieties presented low yield and productivity at 148.1 t/ ha and 157.7 t/ha, respectively. This result was related to the low weight and number of bunches (Figure 5), which might be related to several factors, e.g., difficulty in budburst and low bud fruitfulness in the first few nodes at the base of the shoots (da Silva et al., 2018). According to Camargo et al. (2008), BRS Carmem vines should be pruned to leave six to eight nodes because the first ones are less fruitful. Thus, the pruning management of this variety must be adjusted to increase its potential yield.

### **Biochemical characteristics**

Titratable acidity (TA) range and distribution of medium and late ripening grape varieties are shown in Figure 6. Titratable acidity (TA) range was between 5 g/L – 8g/L, in average of years, the highest titratable acidity in late ripening grape varieties was found in Victory (7.3 g/L) while Moldova (6.0 g/L) had the lowest. Titratable acidity of Husayn kelin barmak (7.3 g/L) was determined also higher than the medium ripening grape varieties while Guzal Kara had the lowest, only 5.3 g/L (Figure 6). The typical titratable acidity (tartaric acid equivalent) (TA) of unripe grapes is around 40 g/L, but this falls to below 10 g/L in ripe grapes. Grape acidity is inversely related to carbohydrate content; in the course of ripening, grape glucose and fructose concentrations increase while acid concentrations fall (Saxton et al., 2009). In present study, titratable acidity was measured all medium and late ripening grape varieties and low concentration.

Based on the established content of sugars and total acids in the grapes of the studied medium and late ripening grape varieties the glucoacidimetric index for each one of them was determined. Its rates were indicative of the grapes quality and its use in winemaking. The calculated rates for 2020 harvest were higher than 2018 and 2019, proving that the grapes were suitable for the production of wines of optimal quality with regard to the chemical composition and the tasting features. For 2020 harvest, the rates were within the range from 2.6 (Taifi Pink and Husayin kelin barmak) to 4 (July), i.e. the grapes not from all varieties, had good enough indicators for the production of quality wines (Yoncheva et al., 2019). Glucoacidimetric index for average of years was from 2.6 (Taifi Pink and Husayin kelin barmak) to 3.7 (July), showing the good characteristics of the grapes to obtain wines with optimal composition and organoleptic profile.



Figure 5. Yield and yield parameters of grape varieties grown on sierozem soil (a) Yield (b) Weight of bunch of grape (c) Number of bunch of grapes



Figure 6. Biochemical characteristics of grape varieties grown on sierozem soil (a) Titratable acidity (b) Glucoacidimetric index

Quantitative data (%) of major carbohydrates such as glucose and fructose, along with the ratio glucose/fructose in the different grape varieties studied, are given in Figure 7. In this study, intervarietal comparison of results showed that the glucose variables were significantly different. Among analyzed samples glucose content of medium ripening grape varieties varied from 6.20% (Guzal Kara in 2018 and 2019) to 10.8% (July and Victory in 2018) (Figure 7). Glucose contents of Victory was determined also higher than the late ripening grape varieties. Guzal Kara and Moldova also lower glucose content among the varieties. Sugar accumulation, especially the concentration of high level of fructose, is a very important physiological process that determines the dessert fruit quality (Kafkas et al., 2006). The mean glucose value of grape varieties was determined as 8.58%. The fructose content in the analyzed varieties was ranged between 8.20 % (July in 2018) and 13.70 % (Chocolate in 2019). According to Average data, Akhtamar (medium ripening variety) and Victory (late ripening variety) were found as highest varieties. July also found the low fructose content. The mean fructose content was determined as 11.04 % in different grape varieties (Figure 7).



Figure 7. Reducing sugars of grape varieties grown on sierozem soil (a) Glucose content (b) Fructose content (c) Glucose + Fructose content (d) Glucose / Fructose ratio

No additional literature is found regarding the glucose and fructose contents of these varieties, therefore the total sugar content of some varieties were compared. Pérez-Magariño and González-San José (2006) determined the sugar content of Cabernet Sauvignon grapes, between 22.5 and 24.4 g/100 in Spain at the two studied vintages. In this study, the glucose and fructose contents of all varieties found to be lower their findings (Figure 7). The glucose and fructose value of Victory was obtained as 21.9 % in this study. Nurgel et al. (2002) found the sugar content of Kalecik karasi as 220 g/L, which is similar to the present result.

The values for glucose and fructose are low with a ratio near one, in grape varieties except with July. The most of the varieties showed that the fructose content higher than glucose content even though the July showed that glucose content almost always had higher than the fructose. Intervarietal comparison of glucose/fructose ratio showed that the July had higher glucose content than the other varieties. On the other hand, Victory had the higher fructose content among the varieties. Varandas et al. (2004) observed the glucose content higher than the fructose content in 5 grape varieties at harvest stage.

# Conclusion

This paper presents recent research highlighting the impacts of climatic factors on droughts and yields of medium and late ripening grape varieties in Turkestan region, Southern Kazakhstan. Drought is one of the major manifestations of climate change in Kazakhstan and exerts a considerable pressure on the agricultural sector, which is the most vulnerable sector to drought in the economy. To mitigate its impact, a warning system to deliver timely information about the possible occurrence of droughts is needed. This is possible only if the influence of large-scale atmospheric processes, formed due to the impact of cosmic-geophysical conditions and interdependent factors in the ocean-glacier-land-atmosphere systems with direct and reverse links, is taken into account. Climatic change is clearly a process determined by the complex interaction of natural and anthropogenic causes. Without explaining the exact mechanism of anthropogenic impacts on the climatic system, we explore the impact of a range of indicators, some of them experiencing clear anthropogenic pressures, to explain the variability in productive and biochemical characteristics of grape varieties grown on sierozem soil in Turkestan region, Southern Kazakhstan. The results can be summarized as follows.

In this study, it has been determined that date of budding in medium and late ripening grape varieties depending on the cultivars and years. According to the meteorological data, the average annual max and min temperatures also increased, and in absolute terms, the change in temperature was 2°C and 3°C, respectively. At the same time, the absolute min temperature decreased by 6°C, from -4 to -10 °C. While climate patterns can differ radically from one year to another (climate variability), climate change is more

concerning, because a significant shift in the long-term climatology would make the wine business unsustainable. Adaptation measures are most probably needed to adapt the current grape varieties to warmer climate conditions and the presence of more frequent and more intense extreme events such as heat waves, heavy rainy or long dry spells.

The grape composition is influenced by climatic conditions and consequently by interannual variability. The 2018 harvest presented better productive and physicochemical grape characteristics. The varieties Husayn kelin barmak and Chocolate presented high fruit yield of grape. Physicochemical grape characteristics were significantly different among the varieties. Husayn kelin barmak and Victory grapes had highest titratable acidity. This study yielded information about the major sugars which are glucose and fructose contents of grape varieties grown in Kazakhstan. The results showed that, there were significant differences in the glucose and fructose content in selected different grape varieties. Akhtamar and Victory were found as highest varieties while July also found the low fructose content. The results reflect that there must be genetic variation among the grape varieties according to their individual sugars.

Even though grapevines have an array of survival strategies, their development is strongly controlled by weather and climate, over a wide range of processes and timescales. Future suitability of a certain viticulture region highly depends on the change on the mean patterns on temperature and precipitation in the coming decades, but also on the impact of extreme temperature and precipitation. For that reason, estimation regional changes in temperature and precipitation and the derived impacts on the suitability of grape cultivation are of paramount importance for the business of gives critical information for making strategic decisions and investments in the near future, which will make the industry more resilient and adapted to climate change.

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