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Research Article

Influence of Inorganic Metals on Antibacterial, Physical and Mechanical Properties of Cement Mortar

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ABSTRACT

Mortars can be exposed to environmental factors and deformations can be observed in the structure after this circumstance. It is thought that using inorganic metal may handle these deformations which harm physical and structural properties. In the present study, cement mortars were produced using different types of metals ((Tin (Sn), Zinc (Zn)) to overcome the abovementioned problem. Afterward, the physical (flowability, UPV), chemical (FTIR), mechanical (compressive and flexural), morphological (SEM-EDX), and antibacterial properties have been investigated. At the end of the study, minor shifts were seen in the Si-O symmetric stretching due to alteration of the C/S ratio. Metals enabled higher mechanical properties by dispersing homogeneously in the matrix. UPV values increased because of metal's conductivity property whereas those metals make it decrease flowability. Also, cement mortar having Zn revealed a higher efficiency against *P. aeruginosa ATCC 2785*. It is thought that the study will contribute significantly to the literature and the industry.

Keywords: *Inorganic metal, cement mortar, antibacterial, mechanical properties*

İnorganik Metallerin Çimento Harçlarının Antibakteriyel, Fiziksel ve Mekanik Özelliklere Etkisinin İncelenmesi

ÖZ

Harçlar çevresel faktörlere maruz kalabilir ve bu durum sonrasında yapıda deformasyonlar gözlemlenebilir. Fiziksel ve yapısal özelliklere zarar veren bu deformasyonların inorganik metal kullanarak giderebileceği düşünülmektedir. Mevcut çalışmada, yukarıda belirtilen sorunları aşmak için farklı metal türleri (Kalay (Sn), Çinko (Zn)) kullanılarak çimento harçları üretilmiştir. Daha sonra fiziksel (yayılma, UPV), kimyasal (FTIR), mekanik (basınç ve eğilme), morfolojik (SEM-EDX) ve antibakteriyel özellikleri araştırılmıştır. Çalışmanın sonunda, C/S oranının değişmesi nedeniyle Si-O simetrik gerilmesinde kaymalar görüldü. Metaller matriste homojen bir şekilde dağılarak daha yüksek mekanik özellikler sağlamıştır. UPV değerleri metalin iletkenlik özelliği nedeniyle artarken, harcın işlenebilirliğini azaltmıştır. Ayrıca, Zn esaslı çimento harcı *P. aeruginosa ATCC 2785*'e karşı daha yüksek etkinlik göstermiştir. Yapılan çalışmanın literatüre ve endüstriye önemli katkı sağlayacağı düşünülmektedir.

Anahtar Kelimeler: *İnorganik metal, çimento harcı, antibakteriyel, mekanik özellikler*

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I. INTRODUCTION

Concrete and mortar are constantly exposed to environmental factors. After this exposure, the pH level of the structure drops from 12 to 9. As a result of this change, construction materials where the concrete or mortar surface is heavily exposed to the water environment such as sewer pipes, marine structures, bridge piers, and pipelines, become more vulnerable to bacteria and fungi.

This circumstance deteriorates progressively and harms the structure. In conclusion, it would not reveal the necessary mechanical strength [1]. Therefore, this problem needs to be eliminated. For that purpose, different materials have been used. One of these materials used is inorganic metal salts [2]. Othman et al. produced cement mortar by using TiO_2 which is the most preferred one against *E. coli* [3]. They observed that improved at early age hydration, porosity reduction as well as enhancement in compressive and anti-bacterial properties. In another study, GuO et al. studied nano TiO_2, as a metal oxide, to determine its antibacterial properties when involved in cement mortar [4]. At the end of the study, they determined that the TiO_2-based mortar revealed inactivation against *E.coli*. However, there has been a trend of looking for new metal types due to TiO_2 aggregation problems in the cement matrix as well as high cost [5]. For that purpose, Singh et al. produced cement mortar by adding ZnO to investigate their antimicrobial properties [6]. They revealed that not only the development of antibacterial properties especially against *E. coli, Bacillus subtilis,* and *Aspergillus niger* but also improved early hydration. Zainul et. al. produced ZnO-based cement mortar to investigate its antibacterial properties [7]. The produced ZnO-based cement mortar revealed activation against some bacteria types with/without UV light. In another study, Klapiszewska et al. produced cement composites containing Lignin/ZnO/Si and examined their antibacterial properties [8]. They determined that the mixtures revealed efficiency against some bacteria and fungi. In addition to the scientific community mentioned above, the comprehensive study related to metal oxide (ZnO) and construction materials to the determination of photocatalytic activity and antimicrobial effects given as a review was carried out by Maria et al. [9]. They stated that ZnO mostly revealed a favorable effect on antibacterial activity, although it varied according to the bacterial species.

In addition to the favorable ZnO effect on bacterial deterioration, Sn plays an important role in industry and is used to investigate its effect on cement properties [10]. Gao et al. used different Sn salts to investigate cement mortar's physical and chemical properties [11]. They determined that the setting time and strength were enhanced by the addition of $SnSO_4$ (stannous sulfate) compared to stannous chloride ($SnCl_2$). Saiko et al conducted a study about the influence of Sn on the hydration of $Ca_3Al_2O_6$ [12]. Results obtained revealed fluctuation in that the value increased in a ratio of 0.5 and 1% Sn, whereas 2% Sn decreased the reactivity of C_3A at the initial period of hydration (3 days). It was also observed that increasing addition of Sn increased the amounts of amorphous phases and hexagonal calcium aluminate hydrates. Although there has been a study related to the Sn effect on cement hydration properties, no study that revealed Sn antibacterial efficiency on the mechanical properties of cement mortars was conducted.

In the coming years, many construction materials will be demolished and rebuilt. Therefore, it is necessary to produce construction materials that have more functional properties and meet the needs. Based on this situation, in the present study, metal-based cement mortars were produced by replacing cement with metals (Sn and Zn).

II. MATERIALS and METHODS

A. MATERIALS

Cement: Ordinary Portland Cement (OPC) CEM I grade and 32.5 R type was used (Beltaş, Duzce, Turkey). Cement analysis (% CaO (64.25), % SiO_2 (18.88), % Al_2O_3 (4.82), % Fe_2O_3 (3.35), % MgO

(1.53), % K_2O (0.01), % Cl^- (0.02), % SO_3(3), Cr_2O_3 (84.69 ppm), % insoluable residue (0.64), % loss ignition (3.35)) was carried out by AKÇANSA (İstanbul, Turkey).

Sand: Calcitic-based fine aggregate sand was used with certain gradation ((2mm–1.6mm (7.07 of %), 1.6mm–1mm (26.26 of %), 1mm–0.5mm (34.34 of %), 0.5mm–0.08mm (32.32 of %)) (Beltaş Düzce, Turkey).

Water: Normal tap water of pH 7.1 was used for preparation of cement mortars.

Metals: Different metal sources ($Zn(NO_3)_2.6H_2O$, and $SnCl_2.2H_2O$) were used in the study as Sn and Zn following provided by Merck (Germany).

Preparation of the cement mortar: Water, cement, and aggregate are prepared in the ratio of 1/2/6 (225/450/1350 w/w). Metals were used in the ratio of 2% by replacing cement. First, the aggregate, cement, metal and water were transferred to mixing bowel and mixed for 60 seconds at 65 rpm in a standard laboratory-type mixer according to TS EN 196-2 (UTEST, Turkey). Those are taken out and mixed 30 seconds manually. Then the mixture were mixed again for 60 seconds in the mixing bowel. After completing the mixing process, the fresh mixtures were poured into prismatic molds (40×40×160 mm) as specified in the standard method and subjected to 20 s vibration in low vibration mode. The mortar specimens were cured in a water bath for 28 days following kept in the mold for 24 hours in the room conditions [13]. The specimens are abbreviated as CM, CMSn and CMZn. The composition ratios of different mortars are given in Table 1.

Table 1. *Composition of different mortars*

Specimen	Cement (g)	Water (g)	Sand (g)	Metal (g)
CM	450	225	1350	-
CMSn	441	225	1350	9
CMZn	441	225	1350	9

After obtaining cured metal-based cement mortars, chemical (FTIR) mechanical (flexural strength and compressive strength), physical (UPV, flowability), morphological (SEM) and antibacterial analysis (disc difusion) were performed.

B. METHODS

For chemical characterization, an FTIR device was used (FTIR, Prestige-21, Shimadzu, Japan) in the 4000–600 cm^{-1} range scan of 20 with a resolution of 4 cm^{-1}. Flexural strength was determined using the three-point loading method in the 0.10 MPa/s loading rate without shocking (Besmak, UTEST, Turkey) [13]. Compressive strength values were determined by measuring halves of the prism broken on flexural testing. UPV analysis was carried out by applying a certain frequencies to transmitting and receiving transducers positioned at opposite ends of cement mortars (NDT, James Instrument, V-Meter MK IV, USA) (180 kHz) to reveal the effect of metals on cement mortar [14].
Flowability revealing the consistency of the mortar was tested using the standard flow table test according to TS EN 1015-3. SEM device was used to monitor morphological alteration of mortars (Quanta 250, FEI, Netherland). Antimicrobial activity was tested against two different microorganisms which are Gram (-) bacteria, *Pseudomonas aeruginosa* ATCC 27853, and Gram (+) bacteria *Bacillus subtilis* NCIB 3610 by using the agar diffusion method following prepared as discs [15]. For that purpose, the inoculated medium was kept at room temperature for 15 minutes, the mortar discs were carefully placed in the middle of the medium and the petri dishes were kept at room temperature for 15 minutes. The prepared petri plates were incubated at 37°C for 24 hours. The zone was measured and determined to evaluate antimicrobial activity.

III. RESULTS and DISCUSSION

FTIR spectra of the produced mortars were taken and the results are shown in Figure 1.

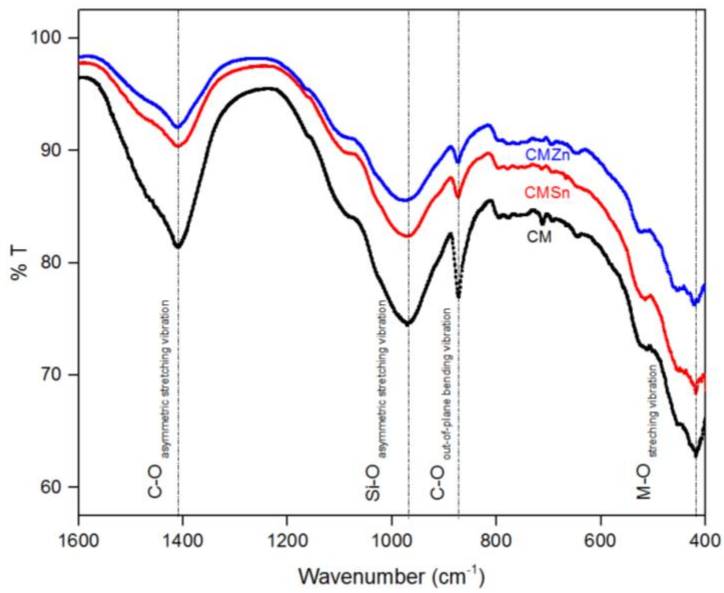

Figure 1. *FT-IR Spectra of Mortars*

The similarities of vibrations in FTIR spectra confirm a similar kind of reaction has been carried out in the cement mortars after incorporating metals. The Si-O symmetric stretching vibration which is one of the main hydration products that is C-S-H gel originating from ordered silicate chains was seen at 969 cm^{-1} for plain mortar (CM) [16]. In those vibrations, there has been a shift after incorporating metals and those vibrations were seen at 976 cm^{-1} for CMZn and 971 cm^{-1} for CMSn since alternating the C/S ratio. Because it is known that asymmetric Si-O vibrations shift to a lower wavelength with an increasing C/S ratio [17].

Another main characteristic vibration is C-O asymmetric stretching vibration and out-of-plane C-O bending vibration which is observed around 1409 cm^{-1} and at 874 cm^{-1}. These vibrations probably belonged to carbonate ions which is attributed to the carbonation of CH by the pozzolanic reaction and can be seen usually as CO_3^{2-} since originated from Na_2CO_3, K_2CO_3, and $CaCO_3$. However, there was no shift in those vibrations. After incorporating metals into cement mortar, there have been some minor alterations in the region of 600-380 cm^{-1}.That region gives a findings related to interactions carried out between metals and inorganic chemicals. The most considerable vibration in that region is Me-O stretching vibration. Those vibration was observed at 419 cm^{-1} for CM [18]. However, after incorporating metals, minor shift was carried out for the CMZn and observed at 421 cm^{-1} while there was no shift for CMSn.

Compressive strength results of the specimen (CM, CMSn and CMZn) is given in Figure 2.

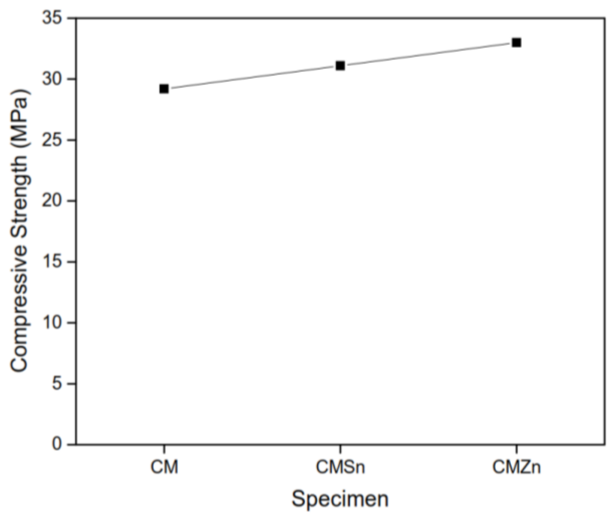

Figure 2. *Compressive Strength of Specimen*

When examining the compressive strength values, the Sn and Zn had a favorable effect on the cement mortar's compressive strengths. While the initial value of the cement mortar was 28.6 MPa, these values increased to 32.8 MPa and 31.6 MPa after incorporating of Zn and Sn, respectively. There has been a similar study carried out by Ślosarczyk et al. that incorporated different metal oxides into the mortar and obtained favorable compressive strength values [19]. Ślosarczyk explained this experimental increment in a different study that might be sourced from Zn and Sn's filling effect that improved mechanically [2]. Dong et al. conducted a similar study on this issue by using five metal types. They stated that higher mechanical properties are observed in metal-based cement mortars [20]. They explained this phenomenon with decreasing porosity.

Flexural strength results are given in Figure 3.

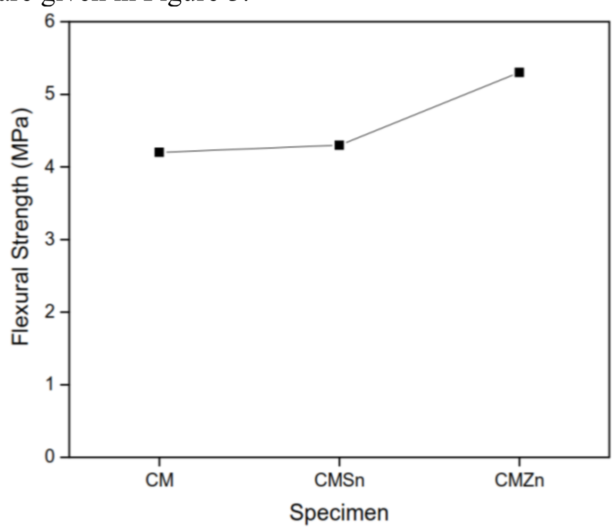

Figure 3. *Flexural Strength of the Specimen*

Figure 4 reveals that flexural strength values improved for both Sn and Zn and the highest results were observed in ZnO-based cement mortar with 5.2 MPa. These circumstances may be explained by the metal's texture structure (pore size and pore volume). However, Klapiszewska et al. conducted a similar study that metals interacting with cement mortars had a lower flexural strength. They explained this circumstance with increasing pore size and filling effect [7]. Therefore, it can be inferred that texture structures and epitaxial properties (the growth of cement hydration products on the aggregate surface and the chemical reaction among metal, aggregate, and cement), influenced the flexural strength.

UPV is applied to obtain information on local voids and defects which gives information about durability and strength properties of cement-based materials. UPV values are given in Figure 4.

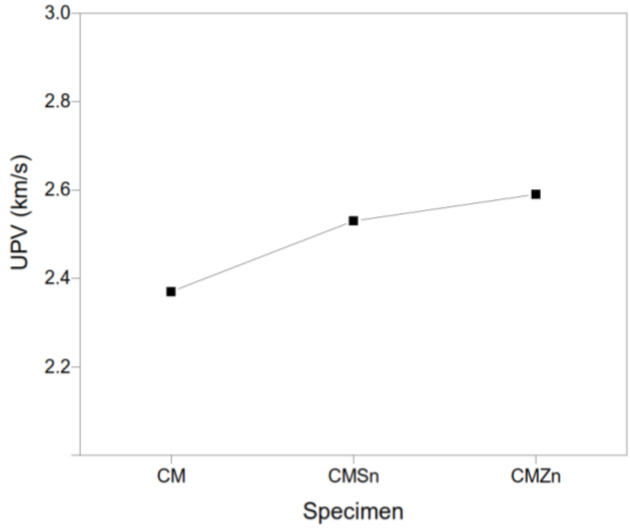

Figure 4. *Ultrasound pulse velocity results of the specimen*

It was seen that metals accelerated UPV values considerable. This situation may be explained by the decrease of voids in the main matrix as supported by SEM [21]. In addition to void decreasing, the high electrical conductivity property of metals may influence velocity and enabled a positive effect on the speed of sound waves [22].
Flowability analysis which reveals workability of the cement mortar is given in the Figure 5 as cm.

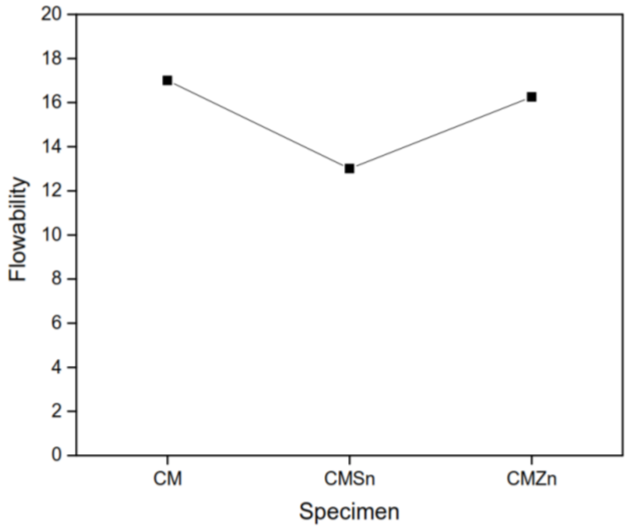

Figure 5. *Flowability properties of the specimen*

It was seen from Figure 6 that flowability values are decreased after interacting with metals. It is thought that these lower values are ascribed in metals prospective properties that metal has hydrate groups in the end of the their chemical structure. Thus, it easily reacts with water found in the matrix when entering metals into the network system. And, this circumstance may decrease the wetting possibility of the cement particles. In addition to the metal structure, the fineness property of the metals may be another reason for lower flowability. It is known that when the fineness of the metals used is smaller than cement, there may be an increased reaction of water compared with cement due to the specific surface area being larger [23]. The observed lower flowability also gives information about early strength property. Thus, it can be inferred that Sn and Zn metals increased the early strength properties of the cement mortar since strength gain is faster than pure cement mortar.

SEM images of pure and metal based-cement mortars were taken to examine their microstructures. The obtained images are given in Figure 6.

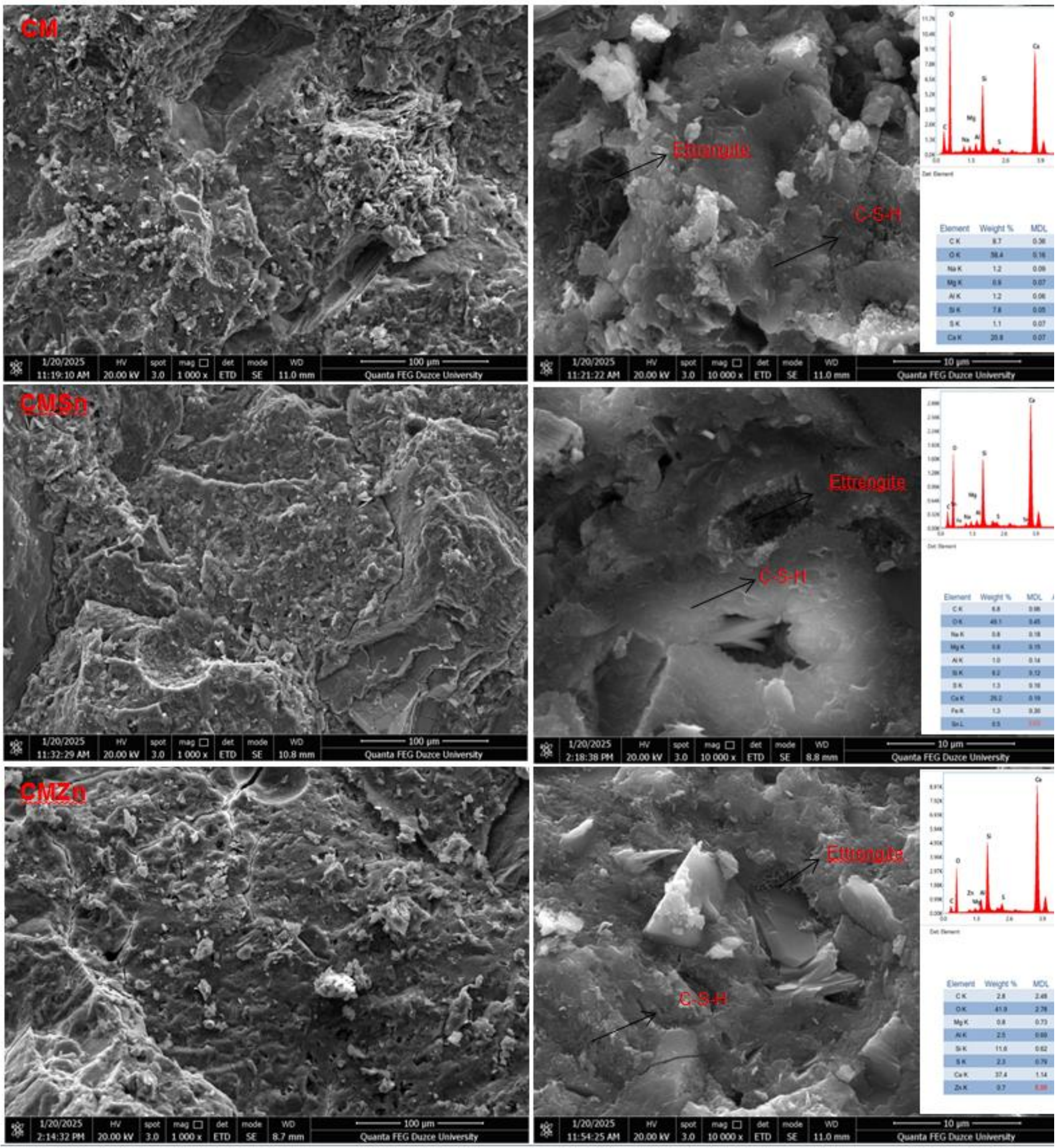

Figure 6. *SEM images (100 µm and 10 µm)*

A dense and dominant microstructure sourced from mainly C-S-H gel, which is the main component of hydrated products forming after interacting water with C_3S and C_2S has been observed as irregular (Figure 6). In addition to the C-S-H main matrix, ettringite structures have been easily seen in the capillary gaps as well as unclustered structures which have mostly fragmented and almost evenly distributed. After incorporating Sn and Zn, the void and pore dimensions started to be lower (especially for Sn) compared to the plain mortar as well as more smoother structure. Therefore, it can be considered that this structural alteration related to the Sn and Zn enabled a filling effect in the matrix. In addition, when the interactions at the interface of the aggregate and the C-S-H main matrix are examined, it was observed that they are in normal appearance. In the EDX spectra which reveal elemental compositions, mainly Ca and Si elements sourced from the C-S-H network were observed in the spectra. The ettringite structure which is a composite of sulfoaluminate that emerges as rod-like crystals was seen as S peak in the spectra.

The antibacterial activity of pure, Zn and Sn-based cement mortar was evaluated against *B. subtilis NCIB 3610* and *P. aeruginosa ATCC 27853* to determine efficacy of metal salts in appropriate nutrient media by using the diffusion method. Results were evaluated with inhibition process. The obtained results of the antibacterial activity of the mortars are shown in Figure 7. Also, zone inhibition measured results are given in Table 2.

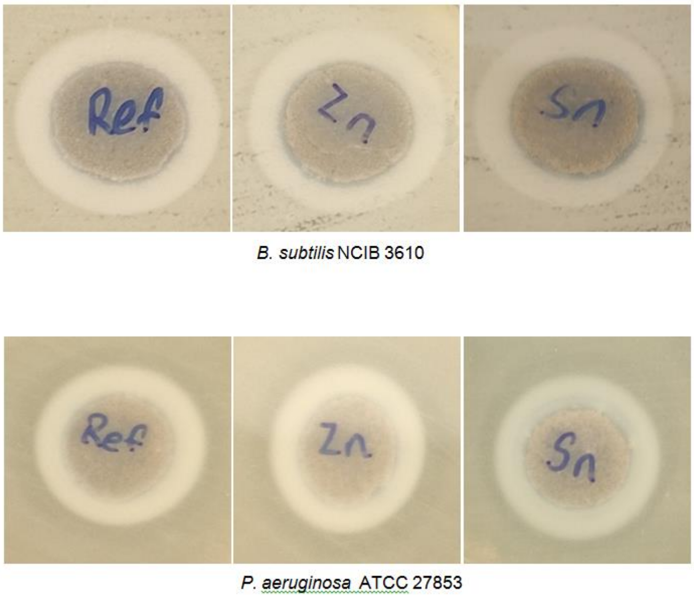

Figure 7. *Inhibition of MeO-incorporated mortars against B. subtilis, and P. Aeruginosa*

Table 2. *Measurement results of zone inhibition (mm)*

	CM		**CMZn**		**CMSn**	
	1	2	1	2	1	2
B. subtilis NCIB 3610	9	9	9	10	8	11
P. aeruginosa ATCC 2785	10	12	12	12	…	…

When Figure 7 and Table 2 are evaluated, it was observed that both Sn-based cement mortar (CMSn) and Zn-based cement mortar (CMZn) revealed no favorable efficacy against *B. subtilis* NCIB 3610 as well as pure cement mortar. However, when investigating the antibacterial efficiency of metal-based cement mortars against *P. aeruginosa* ATCC 27853, the Zn-based cement mortar showed a higher efficacy against *P. aeruginosa* ATCC 27853 compared to that of Sn-based cement mortar. Similar results were determined by Mocioiu et al. that cement mortar having ZnO inhibited the growth of bacterial strains [24].

However, it was observed that pure cement mortar has revealed close activity behavior like Zn-based cement mortar against *P. aeruginosa* ATCC 27853 in the present study. Considering these findings, the produced Zn-based mortar is a little more effective albeit minimally against *P. aeruginosa* bacteria which is found a Gram (-) bacteria type.

IV. CONCLUSIONS

In this study, two kinds of inorganic metals were used to determine their effect on cement mortar's chemical, physical, mechanical, and antibacterial properties by replacing them in a certain ratio of 2% with cement. At the end of the study, it was observed some shifts in Me-O stretching vibrations as well as the main Si-O asymmetric stretching vibration related to C-S-H formed in the network. In the mechanical results, both flexural strength and compressive strength increased with the incorporation of inorganic metals. In addition to the favorable effect on mechanical results, they made it increase the UPV values due to the metal's conductivity property and their texture structure. In the microstructure investigation, the C-S-H structure was dominant, and inorganic metals were distributed homogeneously without revealing any aggregation by decreasing pores. As for antibacterial properties, Sn-based mortars did not reveal any antibacterial resistance against *P. aeruginosa ATCC 27853* and *Bacillus subtilis* NCIB 3610 whereas Zn-based cement mortars revealed minor efficiency against *P. aeruginosa ATCC 27853*. As a result of this study, it can be recommended that inorganic metal-based cement mortars may be utilized in the construction sectors which need higher mechanical properties.
However, it was observed that the inorganic metal ratio in the cement mortar needed to be increased to obtain a higher antibacterial efficiency.

Article Information

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