

Expanded Perlite Mineral As a Natural Additive Used In Polylactide-Based Biodegradable Composites

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Abstract: Polylactide (PLA) is a biodegradable polymer derived from natural resources used in various applications ranging from medical to packaging. In this study, biocomposites were developed by combining perlite mineral (PER), a natural filler material, with a biodegradable PLA matrix in incorporated contaminations of 2.5%, 5%, 10%, and 15%. The purpose of this work is to obtain composites having low production costs while retaining their main properties. Mixing force measurements, tensile, Shore hardness, impact tests, melt flow indices (MFI), and scanning electron microscopy (SEM) evaluations were carried out on composite samples to determine the processing, mechanical, melt flow, and morphological aspects of the developed composites. When the tensile test data were reviewed, minor decreases in the tensile strength and % elongation parameters were noticed with perlite loadings. The inclusion of perlite powder significantly reduced the impact strength value of PLA. Composites with high amounts of PER displayed elevated hardness values. While the MFI results were analyzed, it was deduced that the addition of PER increased the melt flow characteristics of the PLA polymer. At low PER quantities, SEM micrographs displayed that PER particles were homogeneously distributed in the PLA phase. The particle homogeneity in the composite morphology deteriorated as the PER loading ratio in the composites rose. According to the overall results, the highest performance among composites was achieved in the sample including 2.5% PER, and this sample was considered to be the most suitable option for applications regarding PLA-based biocomposite material purposes.

Keywords: Biocomposites, polylactide, perlite, polymeric composites, biodegradable polymer.

Polilaktid Esaslı Biyobozunur Kompozitlerde Doğal Bir Katkı Maddesi Olarak Genişletilmiş Perlit Mineralinin Kullanımı

Öz: Polilaktit (PLA), tıptan paketlemeye kadar çeşitli uygulamalarda kullanılan, doğal kaynaklardan elde edilen ve biyolojik olarak parçalanabilen bir polimerdir. Bu çalışmada, biyokompozitler, doğal bir dolgu malzemesi olan perlit mineralinin (PER) biyolojik olarak parçalanabilen bir PLA matrisi ile %2.5, %5, %10 ve %15'lik ekleme oranlarında harmanlanarak hazırlanmıştır. Geliştirilen kompozitlerin işleme, mekanik, erime akışı ve morfolojik özelliklerini belirlemek için kompozit numuneler üzerinde karıştırma kuvveti ölçümleri, çekme, Shore sertliği, darbe testleri, erime akış indisleri (MFI) ve taramalı elektron mikroskobu (SEM) değerlendirmeleri yapılmıştır. Çekme testi verileri incelendiğinde, perlit yüklemeleri ile çekme mukavemeti ve % uzama parametrelerinde ufak düşüşler görülmüştür. Perlit tozunun dahil edilmesi, PLA'nın darbe dayanımı değerini önemli ölçüde azaltmıştır. Yüksek miktarda PER içeren kompozitler, yüksek sertlik değerleri göstermiştir. MFI sonuçları analiz edildiğinde, PER ilavesinin PLA polimerinin erime akış özelliklerini artırdığı bulunmuştur. Düşük PER miktarlarında, SEM mikrografları, PER partiküllerinin PLA fazında homojen bir şekilde dağıldığını ortaya çıkarmıştır. Kompozit morfolojisindeki partikül homojenliği, kompozitlerdeki PER yükleme oranı arttıkça bozulmuştur. Genel sonuçlara göre kompozitler arasında en yüksek performans %2,5 PER içeren numunede elde edilmiş ve bu numunenin PLA esaslı biyokompozit malzeme amaçlı uygulamalar için en uygun seçenek olduğu değerlendirilmiştir.

Anahtar kelimeler: Biyokompozitler, polilaktid, perlit, polimerik kompozitler, biyobozunur polimer.

1. Introduction:

Minerals are commonly employed as reinforcements for polymeric materials due to their low cost and ease of processing. Perlite is a naturally occurring mineral that is a type of amorphous volcanic silica glass with high water content. When heated, this aluminosilicate can expand 30 times its initially formed volume. Türkiye has the highest perlite deposits, accounting for over fifty percent of the world's total. The other countries with substantial perlite abundance include Japan, Greece, Hungary, Iran, USA, Italy, Mexico, and Iran. In addition to the low-cost plastic additive, coatings, flooring, insulation goods, concrete, lightweight structural components, medicines, dental substances, detergents, soapy products, and water filtering applications have all found uses for perlite

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mineral [1-9]. As a porous volcanic rock, perlite mineral donates weight-reduction in addition to a decrease in production cost as it is used as a filler material [10,11].

Perlite mineral was evaluated as reinforcing material for various polymers in experimental studies according to the literature. In these research studies, perlite inclusions yielded performance improvements for thermal conductivity characteristics in the case of heat storage behavior of poly (ethylene glycol) (PEG) [12], mechanical characteristics of polystyrene (PS) based composites [13], decline in the drug release rate of poly (methacrylic acid) (PMAA) regarding drug delivery application [14]. Additionally, the bone tissue efficiency of hydroxyapatite (HA) was investigated after the integration of perlite [15]. Melt flow properties of acrylonitrile-butadiene-styrene terpolymer (ABS) involving perlite powder were reported in which process parameters were not affected by mineral incorporation related to additive manufacturing application of ABS [16]. The thermal and mechanical performance of poly (vinyl alcohol) (PVA) composites was also optimized with the help of perlite additions [17]. Biodegradable chitosan polymer was filled with expanded perlite which produced composites that showed viscoelastic behavior [18]. Perlite-incorporated polyaniline exhibited a reduction in electrical conductivity compared to unfilled polymers [19]. The flame retardant effect of perlite was also postulated by its integration with polypropylene (PP) composites involving wood flour [20] and expandable graphite [21]. Polyethylene (PE) was compounded with perlite in several research works. In these studies, thermal stability and mechanical strength of composites were reported [22-28]. Thermal insulation behaviors of polyurethane foam (PUF) [29] and epoxy resin [30] loaded with perlite powder were also studied. PLA-based composites involving perlite were fabricated in two research affords according to the literature survey. In one of these studies, perlite addition with high loading amounts (from 20 to 50%) to PLA was evaluated by limited characterization methods including thermal and structural properties [31]. In another publication dealing with PLA/perlite composites, nanosized perlite was used with filling ratios ranging from 1 to 7% by weight. Similarly, the thermal decomposition and crystallinity of composites were reported [32].

Poly lactide (PLA) is a broadly accessible thermoplastic made from materials that are renewable and biodegradable. As a result of these features, polylactide has great potential as a substitution for petroleum-based products. However, in circumstances needing a high level of mechanical resistance, polylactide's strength in its unadulterated condition is frequently inadequate. As a result, a great deal of effort has been expended in researching solutions to this material property defect. The biomedical industry has employed PLA, a biodegradable aliphatic polyester with good qualities for many polymer applications, mostly because of its high cost, which is the result of high-priced polymerization and purifying procedures. The main application area of PLA and related composites is the packaging industry due to its biodegradable character. The use of PLA in textile, transportation, biomedical, and building sectors also exhibits an increasing trend as well as recently being applied as a filament feedstock material in the additive manufacturing process [33-36].

In this work, the PLA matrix was compounded with expanded perlite to achieve low-priced and reduced-weight biocomposites with optimized performances. The resulting composites can be employed as 3D filament material thanks to the wide use of PLA in this application area. The novelty of the study lies in the experimental evaluation of perlite inclusion on mechanical, mixing force, and melt-flow behavior of PLA composites since these properties were not reported in similar research works dealing with PLA/PER composite systems [31,32] as discussed in the literature survey. Melt-blending method was applied to develop composite samples due to its practical integration into high-level production stages in the industry. Mechanical characteristics are crucial factors for PLA as it is used as a packaging material. For this reason, mechanical strength parameters of produced composites were investigated employing tensile, hardness, and impact resistance tests. Reduction of weight for the composite material is highly required in vehicles. The inclusion of a natural mineral in bio-based PLA yields a green composite that retains biodegradable and environmentally friendly behavior. Melt flow measurements were performed since this characterization provides processing ability in the case of developing 3D printed parts via PLA-based composite filaments utilizing additive manufacturing techniques. Besides cost-reduction using low-cost perlite mineral, the force values exerted in the melt-blending process were recorded since it affects the total production cost in industrial manufacturing steps. Morphological analysis was carried out to visualize the dispersion quality of perlite particles in composite morphologies.

2. Experimental

2.1. Materials and instruments:

Expanded perlite with a bulk density of 300–1000 kg/m³ and an average particle size of 40 µm was obtained from Eti Maden, İzmir, Türkiye. The commercial name of PLA polymer was Ingeo Biopolymer which was purchased by Natureworks LLC, USA.

In this study, Xplore Instruments program was employed to quantify force values throughout the extrusion process. The screw force values in the melt were determined using the micro-compounder's rheological software as a function of mixing time. Lloyd LR 30 K universal tensile testing machine was used for tensile properties of composites in accordance with ASTM D-638 standard. Hardness analysis was carried out using an EBP Electromechanical Equipment digital shore hardness tester according to the ASTM D2240 procedure. Coesfeld impact tester was utilized to investigate the impact resistance of composite samples having dimensions of 7.6x2.0x50 mm³ by ASTM D256 standard method. MFI measurements were performed in accordance with ASTM D1238 via Coesfeld Meltfixer LT under the conditions of a 2.16 kg standard load at 190°C. The JSM-6400 Electron Microscope, a field emission scanning electron microscope, was used for observing the morphological characteristics of composite materials. A small coating of gold was applied to the surfaces of the cracked samples from the impact test to establish conductive surfaces.

2.2. Preparation of composites:

Before compounding, PLA pellets and PER powder were vacuum-dried at 80 °C for 6 hours to reduce moisture content. Co-rotating twin screw extruder (Micro-compounder, 15 ml, Xplore Instruments, Netherlands) was utilized for developing composites. In the PLA matrix, PER was compounded by loading ratios of 2.5, 5, 10, and 15 % by weight. Process temperature of 190°C, screw speed of 100 rpm, and mixing time of 5 minutes were applied during the melt-bending process. Using an injection molding equipment (Micro-injector, Daga Instruments, USA), test specimens in the shape of dog bones with dimensions of 7.6x2.0x80 mm³ were prepared. The gauge length of the injection-molded specimens was 50 mm. A barrel temperature of 195°C, a mold temperature of 50°C, and an injection pressure of 8 bar were used in the injection molding process.

3. Results and Discussions:

3.1 Tensile properties of PLA and PLA/PER composites:

The characteristic tensile stress curves versus the percentage strain of PLA and composites were visualized in Figure 1.

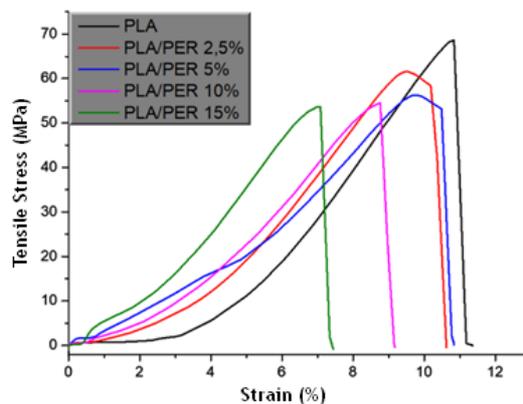


Figure 1. Stress vs. strain curves of PLA and composite samples.

PLA displayed brittle behavior according to its stress vs. strain curve since no necking property at the ultimate strength value. On the contrary, necking behavior was observed for composites filled with low amounts (2.5% and 5%) of PER as curves exhibited a small decline before the breaking point. This finding revealed that PLA/PER composites showed ductile characteristics in low concentrations of PER inclusions. Composites involving high adding amounts (10% and 15%) of PER yielded a brittle tendency similar to unfilled PLA. The necking tendency of a polymer is linked to its ductile property, in which polymer chains resist tensile deformation throughout the test. In this case, PER particles promote the ductility of brittle PLA polymer as they were incorporated with low amounts due to their homogeneous dispersion. On the contrary, weak points in polymer structure were formed for highly-filled PER particles stemming from their poor dispersion in the PLA matrix. The incorporation of PER regardless of its concentration caused the increase in the initial tensile strength of PLA.

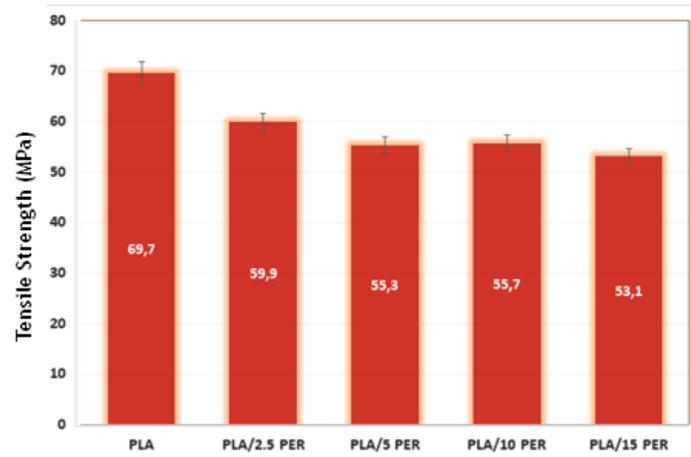


Figure 2. Tensile strength data of PLA and composite samples.

The ultimate tensile strength data of samples were demonstrated as bar graphs in Figure 2. The tensile strength of PLA was reduced by inclusions of PER according to Figure 2. PLA/2.5 PER reached maximum strength value among composites. Further additions of PER caused a slight decrease with a reduction ratio of 7.7% in the tensile strength of composites. Particulate geometry of PER particles might be responsible for this decrease as similar findings were reported in studies dealing with the mechanical performance of PER-filled polymers [13,16,28].

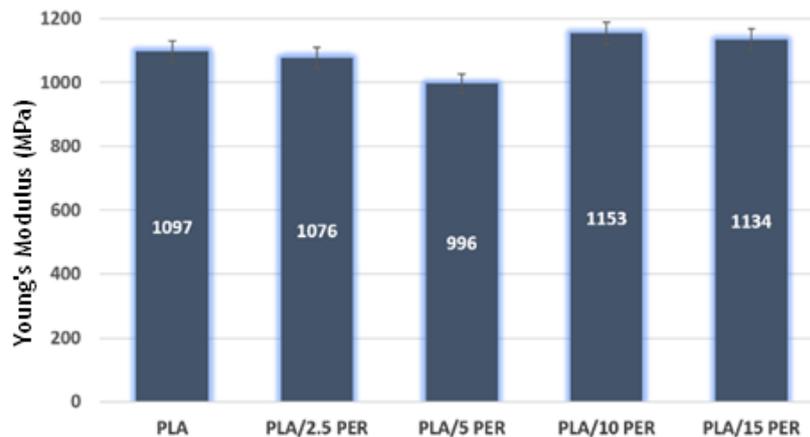


Figure 3. Young's modulus data of PLA and composite samples.

Figure 3 represents Young's modulus values of PLA and relevant composites. PLA/10 PER and PLA/15 PER samples gave higher modulus compared to unfilled PLA where nearly a 5% increase was obtained. This result implied that the addition of high amounts of PER resulted in the improvement of Young's modulus of PLA.

Young's modulus of composites involving high amounts of PER was found to be higher since compounding high amounts of PER increases the hardness and brittleness of the PLA matrix. On the other hand, a small decline was observed for composites containing low amounts of PER. As displayed with elongation data of samples in Figure 4, percent elongation and elongation at break parameters of PLA showed a decreasing trend with PER loadings. Obtaining higher elongation values in composites involving low amounts of PER relative to highly PER-filled composites might be caused by the necking tendency of PLA/2.5 PER and PLA/5 PER as stated in Figure 1.

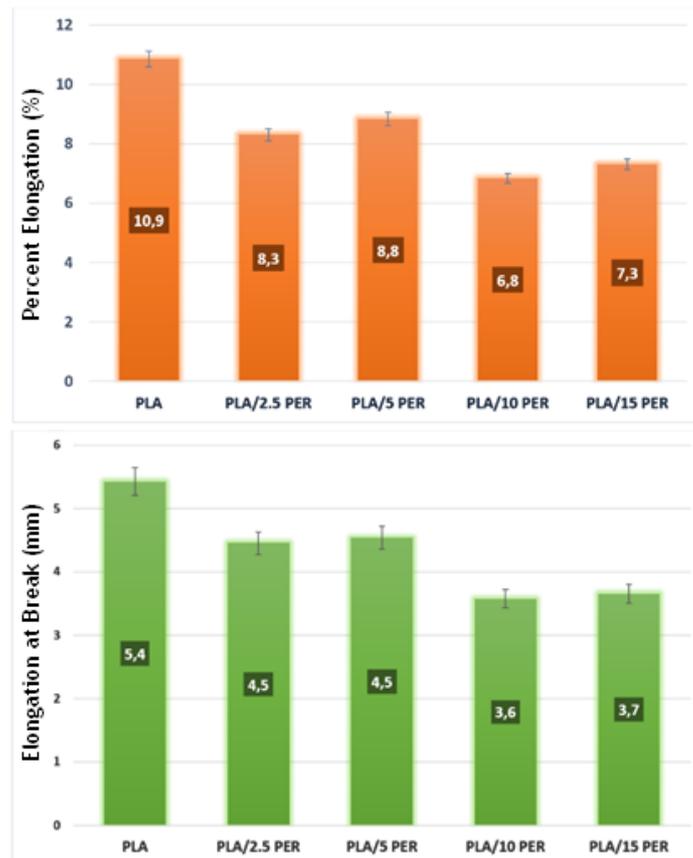


Figure 4. Elongation data of PLA and composite samples.

3.2 Impact resistance of PLA and PLA/PER composites:

The impact properties of PLA and composites are shown in Figure 5. The impact strength of unfilled PLA was reduced proportionally with the increase in the concentration of PER. Lowering in impact resistance was found to be more significant for highly-filled PLA/PER composites where a 31% reduction was calculated. Particulate geometry of PER powder resulted in the propagation of cracks exhibiting a negative effect against impact deformation of the PLA matrix due to the disorientation of load transfer with the inclusion of PER particles. Generally, plate-like additives such as graphite and mica donate impact strength thanks to the high surface area of their layers resulting in the load-bearing capacity during deformation [37-40].

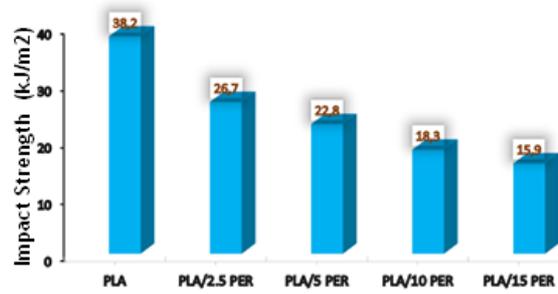


Figure 5. Impact strength data of PLA and composite samples.

3.3 Hardness performance of PLA and PLA/PER composites:

A and D type Shore hardness results of unfilled PLA and PER-involved PLA composites are postulated in Table 1.

Table 1. Shore hardness data of samples.

Sample code	Shore A	Shore D
PLA	95.0±0.1	86.0±0.1
PLA 2,5 PER	95.5±0.1	86.5±0.1
PLA 5 PER	96.0±0.1	88.0±0.1
PLA 10 PER	97.0±0.2	88.5±0.1
PLA 15 PER	97.5±0.1	89.5±0.2

As listed in Table 1, the Shore hardness of unfilled PLA was enhanced with the integration of PER into the composite structure. Hardness values of composites were found to be increased as the PER concentrations were elevated. Higher hardness values were obtained after PER inclusions thanks to the rigidity of the polymer increased with the concentration of mineral additive [34]. According to hardness findings, the correlation between Shore A and Shore D was achieved.

3.4 Force measurements of PLA and PLA/PER composites:

The exerted force values recorded during the melt-mixing process are given in Figure 5. This parameter provides experimental information before production planning related to the fabrication expenditure of resulting composite materials prior to high-scale manufacturing stages.

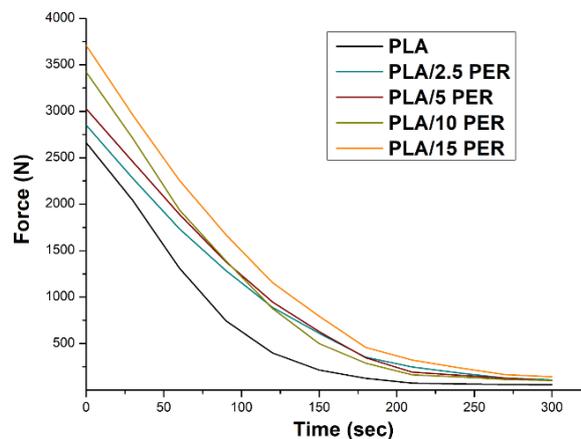


Figure 6. Force measurements of PLA and composites.

As displayed by force versus time curves in Figure 6, PER additions caused an increase in mixing force values since powder inclusions yielded improvement in shear force stemming from enhanced melt viscosity during the extrusion process. Elevated force values were obtained with an increase in the amount of PER due to the aggregation tendency of PER particles in the case of their higher concentrations [41-43].

3.5 Melt-flow behavior of PLA and PLA/PER composites:

In order to evaluate the viscosity of macromolecular melts, melt-flow index analysis is widely applied for thermoplastics. MFI parameters of PLA and relevant composites are shown in Figure 7.

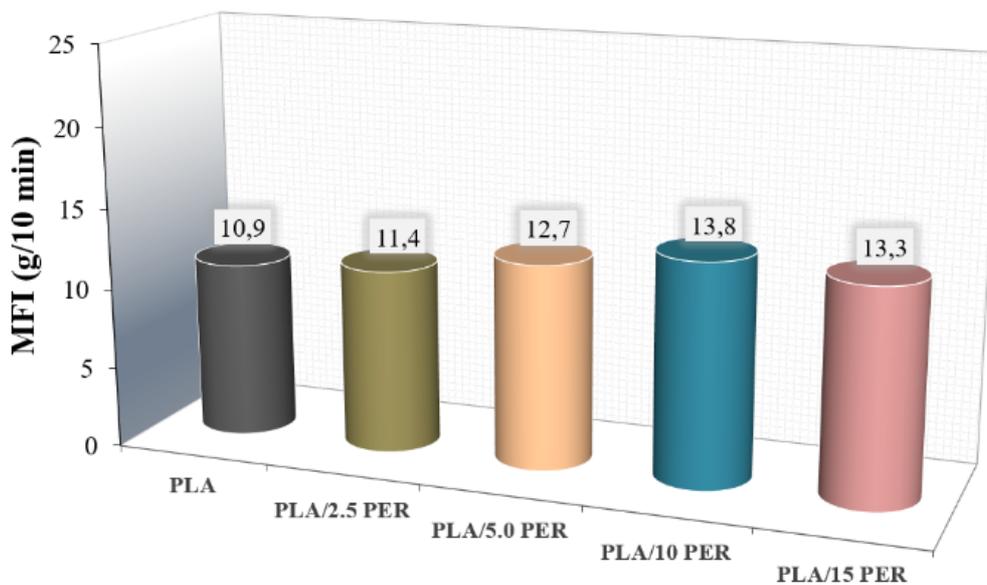


Figure 7. MFI data of PLA and composite samples.

PER-incorporated composites gave relatively higher MFI values compared to unfilled PLA. The increasing amount of MFI was found to be remarkable for composite samples containing higher amounts of PER whereas lower filling ratios of PER displayed very close MFI values to PLA. As an overall observation regarding the melt-flow behavior of PER-loaded PLA composites, MFI parameters of composites were found to be in a narrow range concerning unfilled PLA. This result indicates that PER addition caused no restriction to additive manufacturing application of PLA-based composites as the production of the 3D printing process is considered. The correlation between MFI measurements and additive manufacturing is previously stated in related research work which implied that the printability of polymer is highly affected by its melt-flow characteristics [44-48].

3.6 Morphological analysis of PLA and PLA/PER composites:

Morphological characterization of composite samples was carried out via SEM micro-images as visualized in Figure 8.

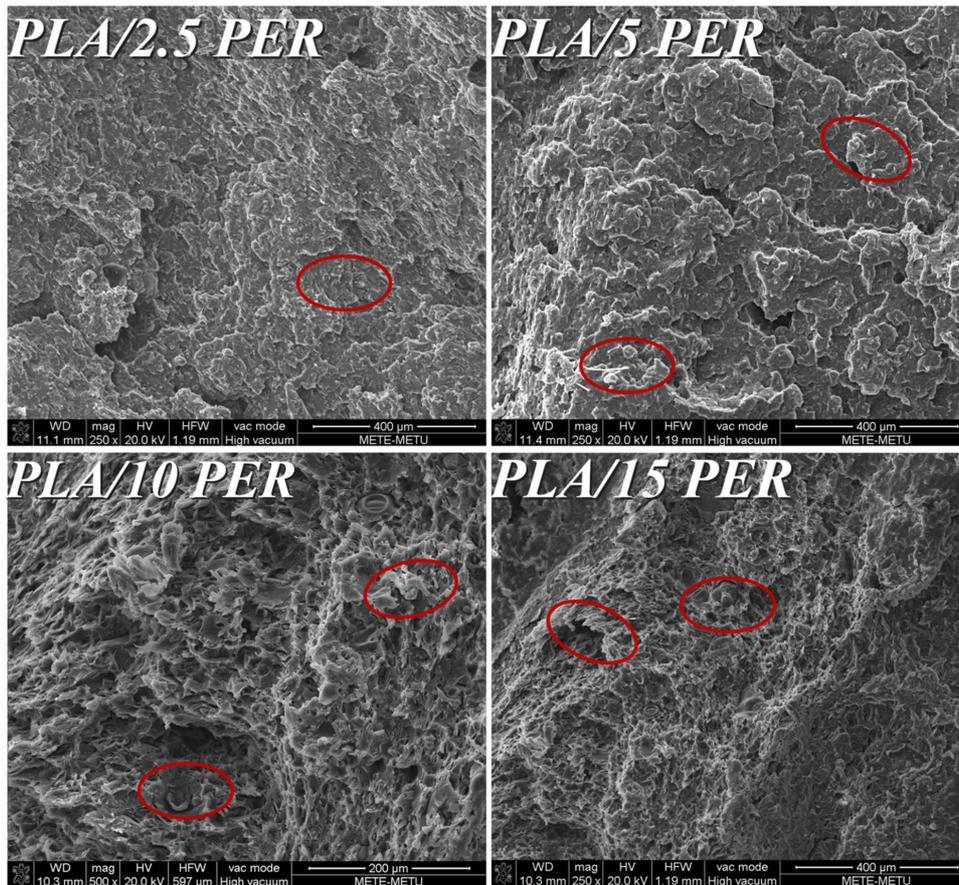


Figure 8. SEM micro-images of composites.

According to SEM micro-images of PLA/2.5 PER and PLA/5 PER samples, PER particles exhibited homogeneously dispersed morphology into the PLA matrix. Conversely, the dispersion homogeneity of PER disoriented as their added content increased due to the tendency for forming agglomeration was favored in the PER phase by particle-particle interactions despite particle-polymer interactions. This finding provided visual evidence for the reduction in related performances of composites described in earlier sections.

4. Conclusion

PLA is a thermoplastic material derived from renewable resources that is considered a green choice for petroleum-derived plastics. Perlite, on the other hand, is a naturally occurring volcanic glass mineral that is frequently used in horticulture, construction, and industrial applications due to its unique properties. While the combination of PLA and perlite offers several advantages, the specific properties and applications of the composite depend on factors such as the perlite ratio of PLA, processing methods, and the intended use of the material. Specifically, in this study, 2.5% PER-added PLA, while exhibiting the highest impact strength compared to other PER-filled PLAs, was considered to be the most suitable option. The properties of PLA and perlite composites can be adjusted by changing the composition and processing parameters. This customizability allows manufacturers to tailor the material to specific application requirements and expand its potential uses. Due to the porous nature of perlite, it can potentially decompose more efficiently than pure PLA, allowing microorganisms to access the material more easily. What's more, it can offer greater sustainability. The use of PLA reduces reliance on petroleum-based plastics and perlite is a naturally occurring resource. The low content of PER is suggested in the case of PLA-based composites since optimum results were obtained for this loading level in this study.

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