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Bazaltik Ponza ile Güçlendirilmiş Poliüretan Elastomer Bazlı Biyo-kompozitlerin Geliştirilmesi

Mehmet YURDERİ[1], Ümit TAYFUN[2], Ahmet BULUT[3*]

Öne Çıkanlar:
- Mineral katkılı elastomer kompozitler
- Mukavemet arttırma amaçlı polimer katkıları

Anahtar Kelimeler:
- Poliüretan Elastomer,
- Biyo-kompozitler,
- Bazaltik Pomza,
- Biyo-medikal,
- Eriyik-harmanlama

ÖZET:

Bazaltik pomza, demir ve magnezyum açısından zengin komatitik tüf bileşimi nedeniyle siyah veya gri bir görünüme sahiptir. Pomzanın gözenekli yapısı, biyomedikal uygulamalar için çeşitli polimer esaslı biyo-kompozit malzemelerin üretimine olanak tanır. Çalışmada, biyo-kompozit numuneler biyo-esaslı elastomerik poliüretan (EPU) matrisine %2.5, %5.0, %7.5 ve %10.0 konsantrasyonlarında bazaltik pomza tozu eklenerek geliştirilmiştir. Bazaltik pomza parçacıklarının yüzey ve element yapısı, SEM/enerji kırınımı X-ışını tekniği kullanılarak incelenmiştir. Biyo-kompozitlerin fiziksel, mekanik, ısısal, eriyik-akış ve morfolojik özellikleri deneysel olarak incelenmiştir. Ponza içeren kompozitler, doldurulmamış EPU ile karşılaştırıldığında, bulgular Shore sertliği ve çekme modülü parametrelerinde artış, çekme uzamada azalma olduğunu göstermiştir. Numunelerin termal çalışmasının sonuçlarına göre, bazaltik pomza eklenmesi EPU'nun ısısal kararlılığında az bir düşüşe ve mekanik deformasyona karşı kararlılıkta bir iyileşmeye neden olmuştur. Ponza yüklemeleri, EPU'nun eriyik akış ve ekstrüzyon torku değerlerini artırmıştır. Bu numunenin taramalı elektron mikroskobu fotoğraflarında EPU matrisinde homojen olarak dağılmış pomza parçacıklarının gözlemlenmesi, %7.5 pomza olan en düşük yükleme oranına sahip EPU numunesindeki kompozitler arasında en yüksek performansı araştırmak için görsel kanıt olarak sunulmuştur. Genel olarak, bazaltik pomza düşük katkı yüzdelerinde EPU biyo-kompozitlerde bir takviye maddesi olarak etkilidir.

Development of Polyurethane Elastomer-Based Bio-Composites Reinforced with Basaltic Pumice

Highlights:
- Mineral-filled elastomer composites
- Polymer additives for improved strength

Keywords:
- Polyurethane Elastomer,
- Bio-composites,
- Basaltic Pumice,
- Bio-medical,
- Melt-blending

ABSTRACT:

Basaltic pumice has a black or gray appearance due to its iron and magnesium-rich komatiitic tuff composition. Pumice's porous structure allows the production of various polymer-based bio-composite materials for biomedical applications. This study developed bio-composite samples by incorporating basaltic pumice powder at 2.5, 5.0, 7.5, and 10.0 percent concentrations into an elastomeric polyurethane (EPU) matrix. The surface and elemental structure of basaltic pumice particles were investigated using the SEM/energy diffraction X-ray technique. The physical, mechanical, thermal, melt-flow, and morphological properties of bio-composites were studied experimentally. Findings showed a rise in Shore hardness and tensile modulus parameters and declined tensile elongation. According to the thermal study, introducing basaltic pumice resulted in a modest drop in the thermal stability of the EPU and an improvement in stability against mechanical deformations. Pumice loadings raised the melt flow and extrusion torque values of the EPU. The observation of the homogeneously distributed pumice particles in the EPU matrix is used as visual evidence to investigate the highest performance among the composites in the EPU sample with the lowest loading rate of 7.5% pumice. Overall, BP is effective as a reinforcing agent in EPU-based bio-composites at low additive percentages.

[1]Mehmet YURDERİ (**Orcid ID:** 0000-0002-0233-8940), Bartın University, Bartın Vocational School, Bartın, Türkiye
[2]Ümit TAYFUN (**Orcid ID:** 0000-0001-5978-5162), Bartın University, Faculty of Engineering, Architecture and Design, Bartın, Türkiye
[3]Ahmet BULUT (**Orcid ID:** 0000-0002-1697-8623), Bartın University, Faculty of Science, Bartın, Türkiye

***Sorumlu Yazar/*Corresponding Author*:** Ahmet BULUT, e-mail: abulut@bartin.edu.tr

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INTRODUCTION

Due to environmental regulations and restrictions, there has been an increasing trend towards ecological composites in recent years. Polymeric bio-composites have advantages such as the practicality of production stages, reusability, lightweight, and low cost. Using natural minerals as an additive in polymeric bio-composites can reduce expenses economically and be environmentally sensitive (Liang, 2013; Çoban and Yilmaz, 2022).

Basic pumice is a dark-colored, brownish-blackish mineral. The most dominant element in the composition of pumice is silica (SiO_2), which varies depending on the region where it is mined. The SiO_2 ratio that pumice contains determines whether it is acidic or basic. While the silica ratio is high in acidic pumice, the content of metal oxides is high in basic pumice. Basaltic pumice is mined in Osmaniye-Adana, Bitlis, Aksaray, Van, Kayseri, Nevşehir, and Mardin in Türkiye (Yazıcıoğlu et al., 2003; Varol, 2016; Öz et al., 2017; Kiliçer, 2023). Pumice was utilized with building components such as concrete, mortar, fiberboard, phase-change, and catalysis-support materials. Weight loss is the primary strategy in these situations, considering the characteristic microporous structure of pumice powder (Yazıcıoğlu et al., 2003; Han et al., 2009; Memiş, 2018; Karaaslan et al., 2022; Koyuncu et al., 2023; Gündüz and Kalkan, 2023).

Numerous research studies have been conducted using pumice as a reinforcement for different polymers. In this approach, blended polyolefins, such as polyethylene (Tayfun and Kanbur, 2018) and polypropylene (Tayfun and Kanbur, 2018; Sever et al., 2019), with pumice, utilizing the melt-mixing technique. This research assessed the mechanical and physical characteristics of composites. Recently, the reinforcing efficiency of both acidic and basic pumice into acrylonitrile-styrene-butadiene 3D filaments was experimentally studied in the case of additive manufacturing applications (Tayfun et al., 2024). Gök et al. and Yılmaz et al. (Gök et al., 2006; Yılmaz et al., 2011) published polyaniline and pumice powder-containing studies. They observed that the inclusion of acidic pumice boosted polyaniline's thermal stability while maintaining conductivity at high temperatures. Integrating of pumice powder into nitrile rubber was evaluated as an alternative reinforcing material instead of silica particles (El-Nemr, 2022). Fleischer et al. and Koyuncu (Fleischer and Zupan, 2010; Koyuncu, 2018) investigated the influence of pumice incorporation on the mechanical and flame-retardant characteristics of solution-blended epoxy-based composites. Based on their results, pumice particles added rigidity and flame resistance to the epoxy matrix. Akkaya additionally reported on the polymer/pumice composite system, demonstrating the viability of uranium and thorium adsorption on pumice-included poly (hydroxy ethyl methacrylate) composites (Akkaya, 2013). Following his findings, pumice-containing composites might be utilized to recover uranium and thorium ions from aqueous solutions. Dike produced biodegradable poly (lactic acid) incorporating pumice material employing a melt-mixing technique (Dike, 2020). In his studies, pumice additions improved the mechanical characteristics of composites. Pumice inclusions enhanced the acoustic and thermal insulation properties of polyurethane-based composites, according to Soyaslan's research (Soyaslan, 2020). Yavuz et al. studied the ability of polyacrylonitrile/pumice composites to adsorb metal ions from aqueous solutions (Yavuz et al., 2008). They demonstrated that pumice-loaded composites may be used as a low-cost adsorbent for removing metal ions from aqueous solutions. Melt-blending techniques were used to combine pumice powder with thermoplastic polymers such as poly (vinyl alcohol) (Jayakrishnan and Ramesan, 2016; Ramesan et al., 2018), poly (β-hydroxybutyrate) (Alvarado et al., 2011), poly (vinyl pyrrolidone) (Ramesan et al., 2016), hydroxyapatite (Koc, 2020) and poly (phenylene sulfide) (Sahin et al., 2014; Sahin et al., 2016), corresponding to the literature survey.

Polyurethane elastomer (Thermoplastic Polyurethane - TPU) is preferred in many application areas due to its high tensile and abrasion resistance, easy processing, and ultimately reusable properties. TPU is used in many application areas, such as footwear, packaging, protective clothing, medicine, cables, and wire and pipe production, especially in the automotive and construction industries.

In addition to phase change materials for use in food packaging, various polymer-based bio-composite materials can be produced in biomedical applications thanks to the porous structure of pumice. In this study, the reinforcing effect of basic pumice in TPU-based bio-composites was investigated. It was aimed to improve the properties of the composites formed after pumice additions, especially mechanical ones. Since basaltic pumice is used as a cheap additive with a lower cost than even TPU the produced composites will be a good option in terms of economic concerns.

MATERIALS AND METHODS

Materials

In this study, bio-based elastomeric polyurethane, which is produced from renewable resources, was used. TPU was purchased from BASF Türkiye, İstanbul via the commercial name of Elastollan® N85A12. Basic pumice was supplied in powder form from Bereketli Madencilik, Adana.

Fabrication of composites

Before the composite preparation stage, TPU and BP were kept in an oven at 100 ˚C for 2 hours in order to remove the moisture in them. Composite samples were prepared using the melt mixing process. BP powder was mixed at 2.5, 5.0, 7.5, and 10 percent by weight into the TPU matrix using a laboratory scale micro-extruder (MC15HT, Xplore Instruments). After the process, composites were obtained in granule form. Composite samples were shaped by injection molding (Daca Instruments) to obtain dog bone-shaped specimens having dimensions of 7.6 × 2.0 × 80 mm^3.

Table 1. Fabrication parameters of composites

Parameter	Value/Unit
Extrusion temperature	200 °C
Mixing speed	100 rpm
Mixing time	5 min
Injection temperature	210 °C
Injection pressure	10 bar
Holding time	1 min

Materials

The particle size of basic pumice powder was analyzed with the Malvern Mastersizer 3000 particle size measuring device. The tensile test is a test performed to measure the strength, flexibility and breaking point of materials. This test determines the strength and durability properties of various materials. In the tensile test, the material is subjected to tension at a certain speed, and the applied stress is measured. This test is performed to determine the stress-deformation behavior of the material. In this study, according to the ASTM D-638 (tensile properties of plastics) standard and with the Lloyd LR 30 K tensile test device, a tensile test was performed on dog bone-shaped composites with a 5 kN load chamber and a speed of 5 cm/min. TPU and its composites were determined using the Shore A measurement Tronic brand Shore hardness device according to the ISO 7619-1 standard. Scanning Electron Microscopy (SEM) is an analysis method used to examine the surface morphology of materials and to observe surface details with high-resolution images. SEM creates an image by sending an intense electron beam to the surface of the composite material and collecting the electrons scattered from the surface of the material. The fracture surfaces of the composite samples obtained from the tensile strength test were examined with a TESCAN brand MAIA3 analytical scanning electron microscope, and SEM images were taken with x1000 magnification. EDX analyzes of pumice powders were also performed using the same device. Thermo-gravimetric Analysis (TGA) is an analysis method used to examine the thermal behavior of materials. This method is used for this research to measure the weight loss of the composite material, especially thermal events such as thermal decomposition, combustion, or separation of the material. During TGA analysis, the sample is heated or cooled along a specific temperature program, and the changes in the weight of the sample are measured. Thermo-gravimetric analysis of TPU and its composites was performed in the 25-600 °C range and at 10 °C/min speed using the Hitachi STA 7300 brand thermo-gravimetric analyzer. It is an analysis method used to examine the flow behavior of molten materials. This method is used to measure the rheological behavior of composite materials. The MFI values of the produced samples were determined using a Coesfeld MeltFlixer device using a standard load of 2.16 kg. The samples cut every 10 seconds were weighed and multiplied by 60 to calculate the MFI values in grams/10 minutes.

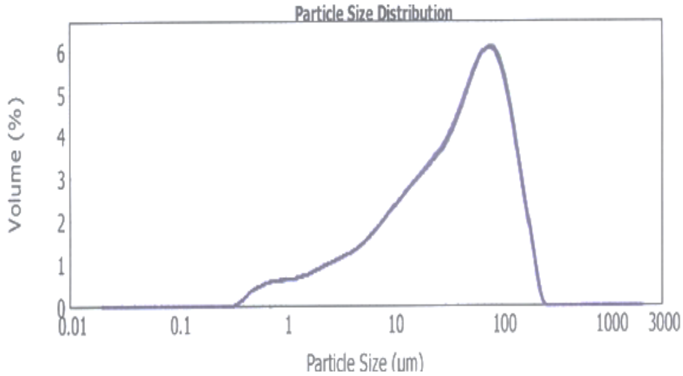
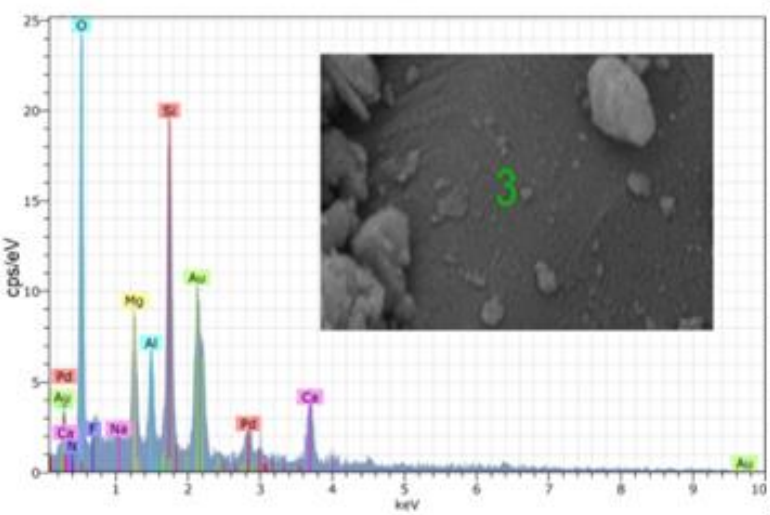


Figure 1. Particle size analysis of BP powder

RESULTS AND DISCUSSION

Characterization of BP powder

According to Malvern analysis displayed in Figure 1, it was observed that basic pumice powder has an average particle size of 60 microns. As a result of elemental analysis with the help of EDX mapping (Figure 2); it was found that basic pumice powder contains mainly SiO_2 with 44.6%. Other contamination of BP includes metal oxides, Al_2O_3, Fe_2O_3, CaO, MgO, and TiO_2, with percentages of 18.1, 9.9, 7.5, 5.7, and 2.1, respectively.



Compound	Composition (%)
SiO_2	44,64
Al_2O_3	18,10
Fe_2O_3	9,94
SO_3	0,06
PbO	-
ZnO	-
K_2O	2,03
MnO	0,13
CuO	-
MgO	5,65
Sb_2O_3	-
BaO	-
P_2O_5	0,71
TiO_2	2,08
As_2O_3	-
CaO	7,47
Na_2O	5,74

Figure 2. EDX elemental analysis of BP

Tensile behavior of composites

Characteristic tensile stress vs. percentage strain curves of TPU and relevant composites are exhibited in Figure 3.

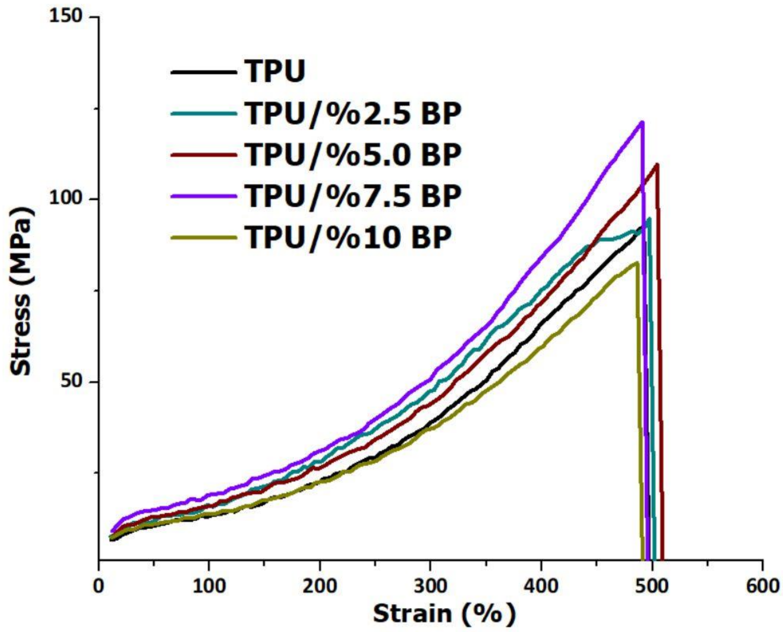


Figure 3. Stress vs. strain curves of TPU and composites

As the composites containing pumice were compared with the unadded TPU, an increase in the tensile stress parameter was observed, while no significant change was detected in the percentage elongation values. The composite sample containing 7.5% basic pumice reached the highest tensile strength value. This concentration was bookmarked as the optimum loading ratio. Further addition of BP caused a dramatic decline in tensile strength. The formation of agglomerated BP particles may be the reason for this sudden reduction. Similar results were achieved in which porous mineral additions led to an increase in the tensile strength of TPU matrix (Sever et al., 2019; Kucuk et al., 2020; Mastura and Noryani, 2022).

Hardness measurements of composites

Shore hardness values of unfilled TPU and composites are listed in Table 2.

Table 2. Hardness data of TPU and its composites

Sample Name	Value (Shore A)
TPU	85.1 ± 0.1
TPU/ 2.5% BP	85.5 ± 0.1
TPU/ 5% BP	85.8 ± 0.1
TPU/ 7.5% BP	86.3 ± 0.1
TPU/ 10% BP	86.9 ± 0.1

*TPU: Thermoplastic Polyurethane, *BP: Bazaltic Pumice

Shore hardness is a characteristic parameter for elastomers and composites. When the values in the table are examined, the addition of BP has an increasing effect on the Shore hardness of TPU. It was observed that the highest addition rate (10%) of BP causes an increase of approximately two units in the hardness of the polymer. These findings indicate that compounding pumice with polyurethane matrix cause no limitations for packaging application of TPU-based composites.

Thermal properties of composites

The thermal resistance of TPU and its composites are examined with the help of TGA and DTG curves as visualized in Figure 4. TPU exhibited a two-step decomposition curve attributed to isocyanate and polyol segments (Barendregt and Van Den Berg, 1980; Petrović et al., 1994; Herrera et al., 2002; Chattopadhyay and Webster, 2009). According to the thermal analysis results of the composite samples, a slight decrease in the thermal stability of TPU was obtained after basaltic pumice additions. High pumice addition rates exhibited a better thermal stability trend compared to low concentrations in terms of thermal decomposition. At the end of the TGA test, an increase in the amount of remaining ash was observed.

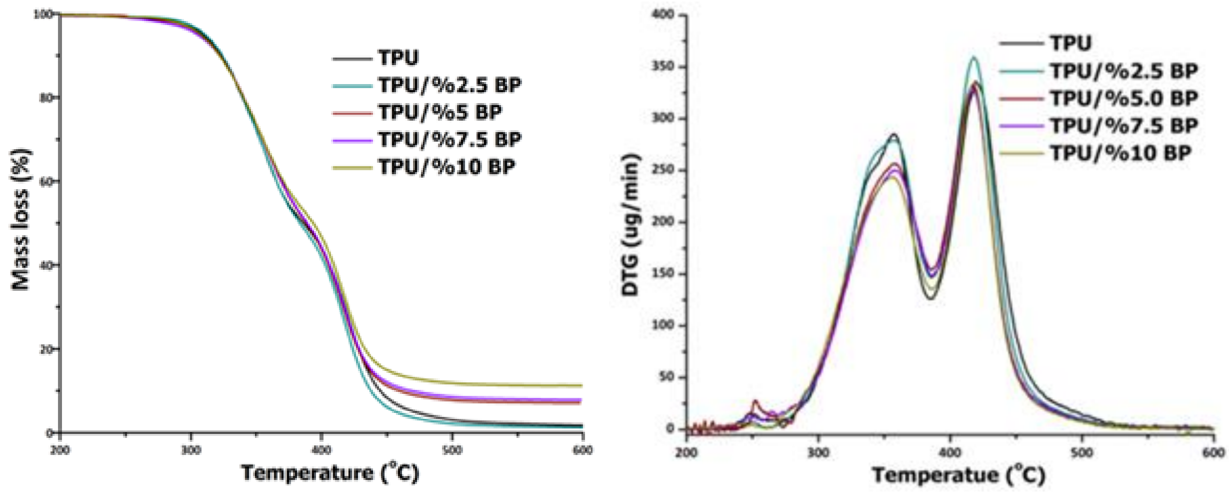


Figure 4. TGA (a) and DTG (b) curves of TPU and composites

Melt-flow behavior of composites

MFI values of unfilled TPU and composites are displayed as bar graphs in Figure 5. The melt flow index (MFI) values of all prepared composites were higher than the MFI value of TPU due to the orientation of pumice particles in the flow direction during melt flow. Pumice loadings caused a significant increase in the melt flow value of TPU. Similar findings were reported, in which pumice inclusions resulted in higher MFI values due to higher specific gravity of BP than polymer (Maierová et al., 2023; da Silveira et al., 2024). MFI test results claim that pumice loadings yield no negative effect for additive manufacturing applications of TPU-based composites.

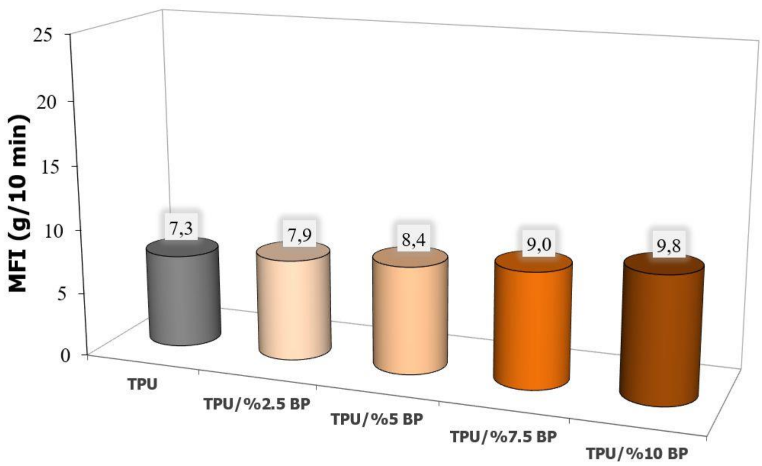


Figure 5. Melt-flow data of TPU and composites

Torque measurement of composites

Torque values recorded during the melt-mixing process are indicative parameters of the production cost of composite materials in the case of their high-scale fabrication stages.

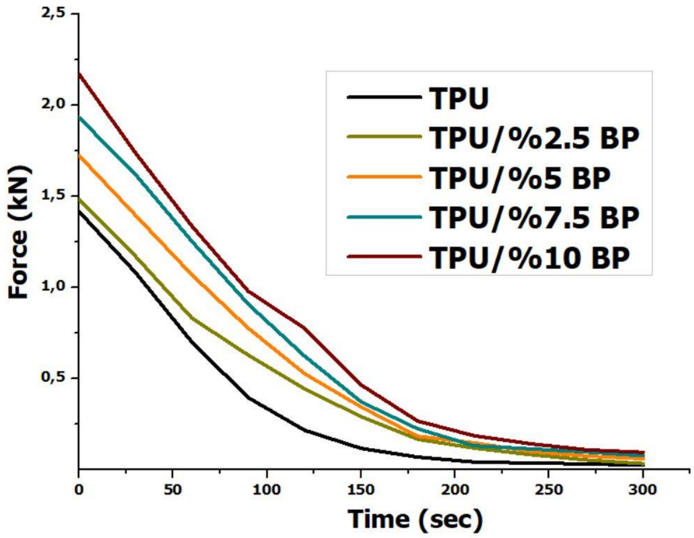


Figure 6. Torque curves of TPU and composites

As the curves of torque values measured in the melt blending process during the preparation of composites were examined against time (Figure 6), it was determined that the mixing force increased in direct proportion to the basic pumice concentration added. Force values revealed that BP inclusions caused the increase in the production cost of composites as the processing steps in large-scale fabrication is considered.

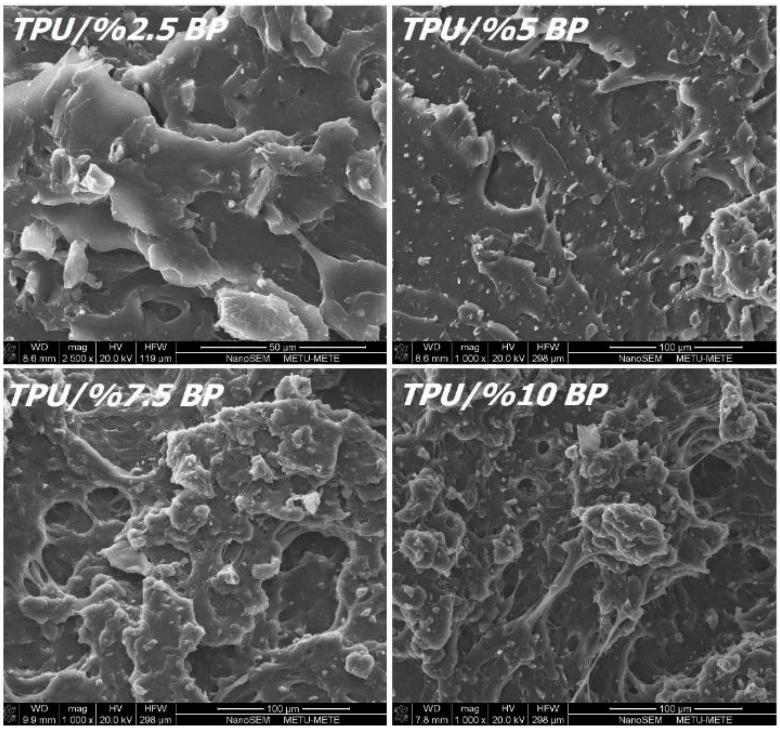


Figure 7. SEM micro-photographs of composites

Morphological characterization of composites

According to SEM micrographs displayed in Figure 7, composite containing 10% BP, a decrease in the distribution homogeneity of the particles in the TPU phase was observed. In a low amount of pumice reinforcements, it was found to be the pumice powders are distributed homogeneously in the composite morphology. Particle-matrix interaction was favored in the case of low loading levels of BP in contrast to highly filled BP, in which agglomeration occurs stemming from the conversion of particle-particle interactions. These observations are consistent with previous results.

CONCLUSION

In this study, four different ratios of basic pumice were blended into the TPU matrix and the effects of pumice addition to the TPU bio-composites on various properties were investigated. According to the results, when the composites containing pumice were compared with the unadded TPU, an increase in Shore hardness and tensile test parameters was observed. Based on the results of the thermal analysis of the samples, a slight decrease in the thermal stability of TPU was detected after basaltic pumice reinforcement. Pumice loadings caused an increase in the melt flow and extrusion torque values of TPU. The determination of the highest performance among the composites in the TPU sample containing 7.5% pumice, which is the lowest loading rate, and the observation of pumice particles exhibiting a homogeneous distribution within the TPU matrix in the scanning electron microscope images of this sample were presented as visual evidence. As a general conclusion, basaltic pumice can be used as a reinforcer in TPU-based bio-composites at low addition percentages. Pumice additions resulted in the expansion of production outcome according to force values recorded during the melt-extrusion process.

Conflict of Interest

The article authors declare that there is no conflict of interest between them.

Author’s Contributions

The authors declare that they have contributed equally to the article.

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