Research Article
Hittite Journal of Science and Engineering, 2023, 10 (2) 177–184
ISSN NUMBER: 2148–4171
DOI: 10.17350/HJSE19030000305

UV and UV+Water Effects on Mechanical, Thermal and Morphological Properties of Thermoplastic Polyurethane Based Composites

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Article History:
Received: 2023/03/04
Accepted: 2023/05/28
Online: 2023/06/30

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ABSTRACT

The degradation times of composite materials containing petroleum-based polymer matrix are quite long in nature and it causes environmental pollution. Researchers focus on producing alternative composite materials with organic and/or inorganic fillers to reduce degradation times in nature. In addition, the mechanical and chemical properties of polymer matrix composites are affected by heat, light, moisture and surface compatibility between matrix and filler. In the light of this information, many researchers examine the mechanical, thermal, morphological and similar properties of organic and/or inorganic filler composites by using artificial aging processes using one or more of the water, UV and temperature parameters.

In this study, bentonite with surface modification by epoxysilane agent, and bentonite without any surface modification will be added to the thermoplastic polyurethane (TPU) polymer matrix, which has two different segments, and will be mixed in a twin screw extruder and it will then be shaped by the injection molding process. Artificial aging processes of the obtained composites will be done under UV and UV+water for 2 and 4 weeks, respectively. The reason for choosing UV and UV+water processes is that UV and water processes have not been applied together at the same time and there is not much knowledge in the literature on this subject. Mechanical, thermal and morphological properties of composites were investigated.

Keywords:
CTPU, Bentonite, Artificial aging, Silanization, Composite

INTRODUCTION

Along with consumer demands and expectations, interest in environmental sustainability issues continues to increase, and technological developments also increase demands for global resources (1–3).

Amount of petroleum-based synthetic plastic products are decreased because of the oil crisis. Nowadays, polymers with inorganic fillers were started to attraction, and they have begun to become engineering materials with a wide variety of properties (1).

Organic or inorganic filler-added polymer composites, which have become increasingly common in engineering applications in recent years, can be produced at both laboratory and industrial scale and can be obtained at low cost (4).

Thermoplastic polyurethane (TPU), which is easily synthesizable from laboratory scale to industrial production lines, has a hard segment consisting of highly polarized groups such as low molecular weight glycols or diamine reacting with diisocyanate and reinforced by crosslinking, and a soft segment consisting of polyester and polyether units; attracts the attention of researchers and manufacturers (5). Thermoplastic polyurethanes are preferred in artificial tissues and organs, biopharmaceuticals, fireproof applications and packaging materials, thanks to their advantages such as biocompatibility, rubbery elasticity and easy processing. (6).

Bentonite is an inexpensive, environmentally fri-

Cite as:
Sismanoglu S. UV and UV+Water Effects On Mechanical, Thermal and Morphological Properties of Thermoplasticpolyurethane-Based Composites. Hittite J Sci Eng 2023;10(2):177-184. doi:10.17350/hjse19030000305

endly, easily accessible layered silicate clay containing biotite, quartz-containing smectite and a small amount of feldspar (4, 7). Thanks to the presence of OH groups in its structure, it allows the adsorption of heavy metal ions and organic compounds, allowing it to be used as a good adsorbent on environmental pollution (4). However, since OH groups make the structure more hydrophilic, it causes incompatibility problems with the polymer matrix with hydrophobic character (4, 7).

The properties of polymer composites vary depending on the polymer matrix/fill interface compatibility, factors such as temperature, humidity, sunlight, and radiation (8). Surface incompatibility problems caused by the hydrophilic character of the fillers and the hydrophobic nature of the polymer matrix can be minimized by surface modification processes such as alkalization, silanization and acetylation (2). Mechanical and chemical effects are also the main cause of polymer degradation (9–12).

It is common knowledge that different environmental conditions affect the mechanical properties of TPU-based composites. As reported in the literature various physicochemical and mechanical changes occur (10, 13–16). The degradation process of polymers is affected by many parameters, especially aging time, immersion time and chemical properties. (9, 17–22).

Examination of aging processes in polymers is important in terms of examining the mechanical and chemical behavior of polymers against heat, light, water and radiation and providing information about their degradation. For this reason, it is examined how the properties of polymer materials change with artificial aging by making changes such as UV, water and temperature. Accelerating the aging process with aging conditions in laboratory environments in accordance with outdoor conditions is called artificial aging. Xenon arc or Hg lamp artificially creates the effect of UV light, causing partial degradation of the polymer (23). Apart from UV, the effect of artificial aging systems established with temperature and water on composite materials can be divided into three aspects. First, it can cause damage to the polymer matrix, reinforcing fibers and the interface between them. Second, temperature change can be a source of thermal stress damage. Third, moisture absorption can cause crack growth by plastification of the matrix and damage to the crystal structure (8, 24, 25).

In general, an increase in temperature causes an increase in the diffusion rates of moisture (10, 26–28). Hollande and Laurent (19) investigated the effect of water on the degradation of thermoplastic elastomer polyurethane-coated polyurethane. Aglan et al. (9), was found that how to effect the mechancal performance of elastomeric polyurethane after applying hydrothermal aging.

In this study, the composites obtained by adding the applied bentonite to the TPU polymer matrix with and without surface modification; with the application of UV and UV+Water artificial aging processes at room temperature and without any aging process, information will be obtained about how mechanical, thermal, and morphological properties change. With this study, information will be gained about the aging processes of not only pristine TPU based polymer composites examined in the literature, but also polymer composites with fillers. In addition, artificial aging systems, in which the effects of temperature and humidity are examined simultaneously, are generally used in the literature. In this study, information about the aging performed by using UV and water together at constant temperature will be obtained.

MATERIALS AND METHODS

Chemicals and materials

Bentonite (BE) which was used as filler, was purchased from Yurtbay Seramik, Istanbul, Turkey. TPU (Pearlthane® ECO D12T85) is the polymer matrix and biomass ratio of TPU is 46%, was supplied from Lubrizol Advanced Materials Spain SL (FKA Merquinsa). Silanization agent is [3-(2,3-epoxypropoxy)-propyl]-trimetoxysilane (ES) (Alfa Aesar (Haverhill, MA, USA)). Ethanol is used in the silanization process as a solvent was purchased from Merck (MerckKGaA, Darmstadt, Germany).

Surface modification of bentonite

The silanization process was performed as the previous studies using (2, 29, 30). The concentration of the silaning solution is 2% by weight. Unmodified sample is called BE, where the modified sample is named as ES-BE.

Preparation of composites

Pristine TPU and TPU/BE samples were mixed with twinscrew micro-extruder (MC 15HT, Xplore Instruments, Sittard, Holland) and shaped in the injection molding device (Microinjector, Daca Instruments, Santa Barbara, CA, USA) as a previous studies procedure (2, 29, 30). Ratio of filler (BE) were the constant (30%) all of the samples.

Artificial aging process of composites

The samples to be subjected to UV artificial aging were kept under Hg lamp for 2 and 4 weeks at room temperature. After weighing the composite samples to be subjected to UV+water artificial aging, they were placed in containers containing equal amounts of water and kept under Hg lamp for 2 and 4 weeks at room temperature.

Instrumentation

FTIR/ATR analysis of both unmodified and modified BE powders, tensile and Shore A hardness, thermal analysis tests of composites were performed as our previous studies (29, 30). Also, SEM micrographs were taken as our previous studies, too (29, 30). SEM magnifications were taken at x 2000 and x 10000.

RESULTS AND DISCUSSION

Structural characterization of bentonite powders

The FTIR/ATR graph of pristine and epoxysilane modified bentonite is shown in Figure 1. In addition, the comparison table of the observed peaks and the peaks specified in the literature is given in Table 1.

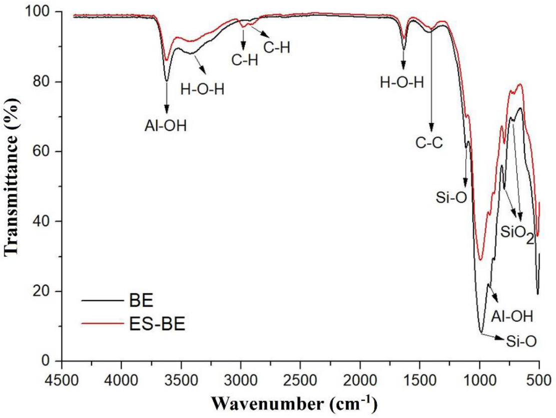

Figure 1. FTIR/ATR of Bentonite and epoxysilaned bentonite.

Table 1. Observed peaks and literature-reported peaks for bentonite.

Observed peaks (cm^{-1})	Vibration mode	Literature reported peaks (cm^{-1})
3627	Stretching vibration of Al-OH in silicate	3619 (31)
3420	H-O-H stretching and bending vibration of silicate layer	3391 (31)
2988	Asymmetrical C-H stretching of $-CH_2$ group	2927.61 (32)
2900	Symmetrical C-H stretching of $-CH_3$ group	2871.68 (32)
1634	H-O-H bending for water molecules	1635 (33)
1435-1388	C-C stretching vibration in aromatic	1456.09 (32)
1110	Perpendicular Si-O stretching	1108 (33)
990	Si-O bending vibration	990 (33)
912	Al-OH founded	915 (33)
794	Presence of quartz and free (amorphous) silica admixture	789-787 (33)
711	Presence of quartz and free (amorphous) silica admixture	689-687 (33)

As seen in Figure 1, the peak observed at 3627 cm^{-1} and caused by the stretching vibration of Al-OH in the silicate decreased with the application of surface modification with epoxysilane to the bentonite (31). While the H-O-H stretching and vibration peak absorbed in the silicate layer at 3420 cm^{-1} (31) is more rounded and larger in pristine bentonite, it was observed that this peak decreased with the increase of hydrophobicity of the surface in epoxysilane surface modified bentonite. It has been proven that the modification of pristine bentonite with epoxysilane is successful with two new peaks formed at 2988 and 2900 cm^{-1}. Among these peaks, the peak observed at 2988 cm^{-1} is due to asymmetrical C-H stretching in the $-CH_2$ group (32), and the peak at 2900 cm^{-1} is related to the symmetrical C-H stretching in the $-CH_3$ group (32). In addition, H-O-H bending for the water molecule caused a decrease in the peak intensity in the epoxysilanizated bentonite sample, which shows that the surface modification process increased the hydrophobicity and was successful (33). The peak formed in the range of 1435-1388 cm^{-1} is an indicator of the epoxy group and while this peak is not observed in pristine bentonite (32); emerged by forming a new peak in the bentonite sample that had surface modification with epoxysilane. Specific vertical Si-O stress of bentonite (33), Si-O bending vibration (33), Al-OH formation (33), presence of quartz and free (amorphous) silica additive (33) peaks at 1110, 990, 912, 794, and 711 cm^{-1}, observed, respectively and the intensity of these peaks decreased after epoxysilanization.

Thermal properties of composites

Figure 2 shows the TGA curves of pristine TPU (a), unmodified bentonite added TPU based (b) and surface modified bentonite added TPU based composites, respectively. Pristine TPU and composites have two thermal decomposition stages at 280°C and 460°C due to urethane bonds in the hard TPU segment and polyol groups in the soft TPU segment (34, 35). It showed that TPU and its composites corresponded to the oxidation of polyethers with lower thermal stability in the first stage of the

TGA curves (260-480°C), while in the second step above 480°C, the polyethers and polyamide fractions showed simultaneous thermal and oxidative degradation (36).

As seen in Figure 2a, the thermal stability of the unaged pristine TPU is lower than that of the artificially aged samples under UV and UV+water conditions for 2 weeks in the first step. In the second step of thermal decomposition, the thermal stability of the unaged pristine TPU is lower than the sample with 2 weeks of UV+water artificial aging, while it is higher than the sample that only UV has been applied for 2 weeks. When we look at the 4 week artificial aging conditions, it is seen that UV and UV+water artificial aging conditions reduce the thermal resistance of pristine TPU both in the first and second stages. Looking at the UV and UV+water 4-week data, it was seen that both artificial aging conditions affected the thermal stability of pristine TPU almost equally.

When we look at the TGA curve of bentonite-added TPU-based composites (Figure 2b), it was found that the sample with the lowest thermal resistance in the first and second stages was applied for 2 weeks with UV+water aging condition. The thermal stability of the samples in the first step without aging, only UV applied for 2 weeks and 4 weeks, and UV+water applied for 4 weeks were almost close to each other. In the second step, it was observed that the thermal resistance of the sample, which was only UV applied for 2 weeks, was slightly higher than the other samples.

According to Figure 2c, when the composites containing bentonite powders with surface modification with epoxysilane were examined, it was observed that the thermal resistance of the sample, which was treated with UV+water artificial aging for 4 weeks in the first step, was the lowest, while the thermal resistance of the sample that was only UV applied for 2 weeks in the second step was the lowest.

In the second stage of thermal decomposition of unmodified and modified bentonite added TPU-based composites, the thermal stability of the samples that were treated with both UV and UV+water artificial aging for 4 weeks were almost the same, the mass loss was less than the pristine TPU without aging, and higher thermal stability. stability was observed.

It was observed that the mass losses seen in Figure 2b and c were caused by the evaporation of desorbed water molecules adsorbed on the cations in the bentonite interlayer below 200 ˚C. Above 350 ˚C, mass loss occurred by removing water molecules from the crystal lattice with an alumina octahedral sheet sandwiched between two silica tetrahedral sheets. In addition, small mass losses occurred with the dehydroxylation of bentonite at higher temperatures (37).

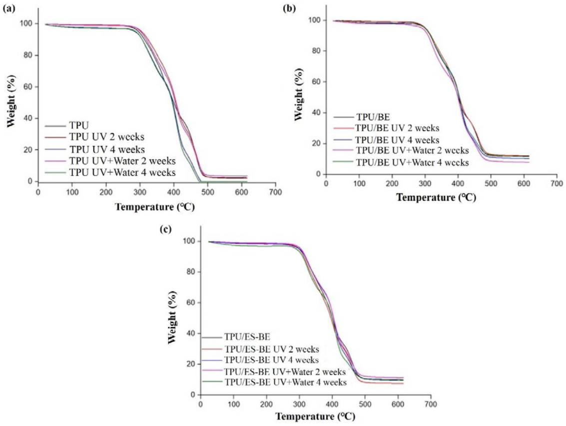

Figure 2. TGA of TPU composites in different aging conditions (a), TPU/BE composites in different aging conditions (b), TPU/ES-BE composites in different aging conditions (c).

Mechanical properties of composites

Figure 3 shows the tensile properties of pristine TPU (a), unmodified bentonite added TPU based (b) and surface modified bentonite added TPU based composites, respectively. In Table 2, the tensile strength, elongation at break, Young's modulus and Shore A hardness data of TPU, TPU/BE and TPU/ES-BE composites under various aging conditions are given.

When we look at the tensile results of pristine TPU and TPU/ES-BE composites, it was seen that the tensile strength of the sample that was not subjected to any aging treatment was lower than the samples that were only UV aged for 2 weeks and higher than the samples that were only UV aged for 4 weeks. In the light of these results, it has been seen that in cases where UV aging time is short, it is related to structural and molecular reorganization, and with the increase of aging time, chemical changes are dominant and it is related to degradations such as oxidation and chain scission in the polymer (8, 38, 39). UV exposure time affect the mechanical properties of composites because it causes the a lot of chain scission reactions (38, 40, 41). They showed that the elongation and ultimate tensile strength decreased as the exposure time to UV increased (38).

When we look at TPU/BE composites, it is thought that as the UV exposure time increases, additional crosslinking is revealed and this crosslinking leads to an increase in tensile strength (38). The results are in good agreement with those reported in several articles (42–46). Subocz et al. (43) and Claude et al. (42) showed that the long-term effect of UV mainly consists of additional cross-linking to form new high-order supramolecular structures, thus explaining the reason for the increase in Young's modulus of the UV-

Table 2. Mechanical properties of composites.

Samples	Tensile Strength (MPa)	Elongation at break (%)	Youngs' modulus (MPa)	Shore A
1	32.26 ± 0.9	562.61±0.2	115.02±0.3	52
2	36.30±0.3	737.32±0.7	79.54±0.6	48
3	29.24±0.4	507.18±0.6	80.77±0.9	51
4	34.24±0.1	656.14±0.5	115.15±0.8	49
5	38.29±0.4	716.62±0.2	125.34±0.6	48
6	33.87±0.7	598.12±0.1	119.27±0.5	49
7	34.26±0.9	593.53±0.5	130.21±0.2	49
8	34.34±0.3	606.81±0.7	153.50±0.6	49
9	35.30±0.1	624.55±0.9	133.92±0.9	53
10	33.20±0.8	651.87±0.2	141.92±0.5	46
11	36.12±0.7	634.79±0.4	149.02±0.1	46
12	35.28±0.5	632.75±0.1	134.65±0.9	50
13	32.45±0.2	583.69±0.8	94.06±0.6	47
14	35.28±0.4	645.30±0.4	135.51±0.6	51
15	35.76±0.8	650.34±0.7	94.13±0.1	51

*1:TPU (Pristine), 2:TPU (UV 2 weeks), 3:TPU (UV 4 weeks), 4:TPU (UV+Water 2 weeks), 5:TPU (UV+Water 4 weeks), 6:TPU/BE (Pristine), 7:TPU/BE (UV 2 weeks), 8:TPU/BE (UV 4 weeks), 9:TPU/BE (UV+Water 2 weeks), 10:TPU/BE (UV+Water 4 weeks), 11:TPU/ES-BE (Pristine), 12:TPU/ES-BE (UV 2 weeks), 13:TPU/ES-BE (UV 4 weeks), 14:TPU/ES-BE (UV+Water 2 weeks), 15:TPU/ES-BE (UV+Water 4 weeks).

exposed polymer (38).

In TPU, TPU/BE and TPU/ES-BE composites, all of the samples that were treated with UV+water aging for 2 and 4 weeks (except for TPU/BE (UV+water 4 weeks)) had higher tensile strengths than those that were only UV treated for 2 and 4 weeks. In the light of these results, it was observed that the formation of cross-linking was more in the samples with UV+water aging process (38). It was also seen that the penetration of water into the polymer structure was important in the samples with UV+water aging at various times, and the diffusion of water molecules, especially in the amorphous phase, increased the flexibility of the polymer chains (8).

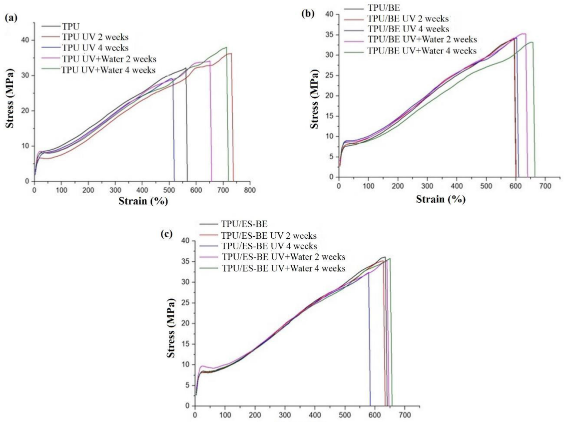

Figure 3. Tensile graphs of TPU composites in different aging conditions (a), TPU/BE composites in different aging conditions (b), TPU/ES-BE composites in different aging conditions (c).

According to the hardness value results of TPU, it was determined that the Shore A hardness value of the unaged sample was the highest and the hardness values were close to each other after the aging processes.

In TPU/BE composites, on the other hand, the hardness value of the unaged sample was found to be the same as the samples that were only UV aged for 2 and 4 weeks. It was observed that the hardness value of the sample decreased as a result of UV+water aging for 4 weeks, but the hardness value of the sample was the highest with 2 weeks of UV+water aging process.

It was observed that the hardness values of the samples increased after various aging processes were applied in TPU/ES-BE composites, and the hardness values were the highest and equal to each other with UV+water aging process for 2 and 4 weeks.

Morphological characterization of composites

Looking at the SEM micrographs of pristine TPU (Figure 4), it is seen that the surface of the unaged sample is smoother. As a result of aging processes, it was observed that the surface roughness of pristine TPU samples increased. With the effect of UV aging, it caused the cross-links to break in the hard segments of TPU and the formation of voids (47). It was observed that as the UV application time increased, the voids increased with the increase in the breaking rate of the cross-links. In the same way, it was observed that the cracks increased and the voids increased in the pristine TPU with the increase of the combined application time of UV and water in the samples applied UV+ water aging process.

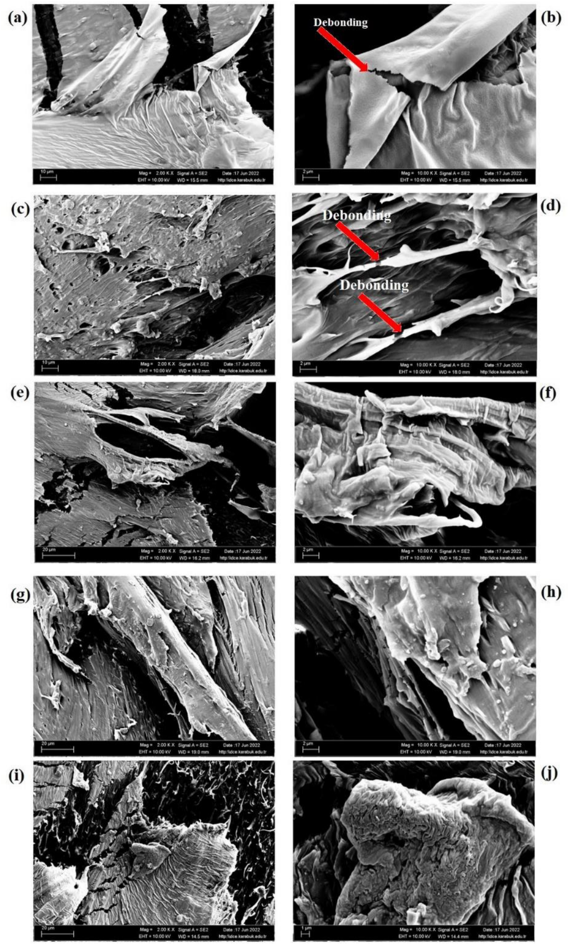

Figure 4. SEM micrographs of unaged TPU composites (a-b), aged by UV in 2 weeks (c-d), aged by UV in 4 weeks (e-f), aged by UV+water in 2 weeks (g-h), aged by UV+water in 4 weeks (i-j).

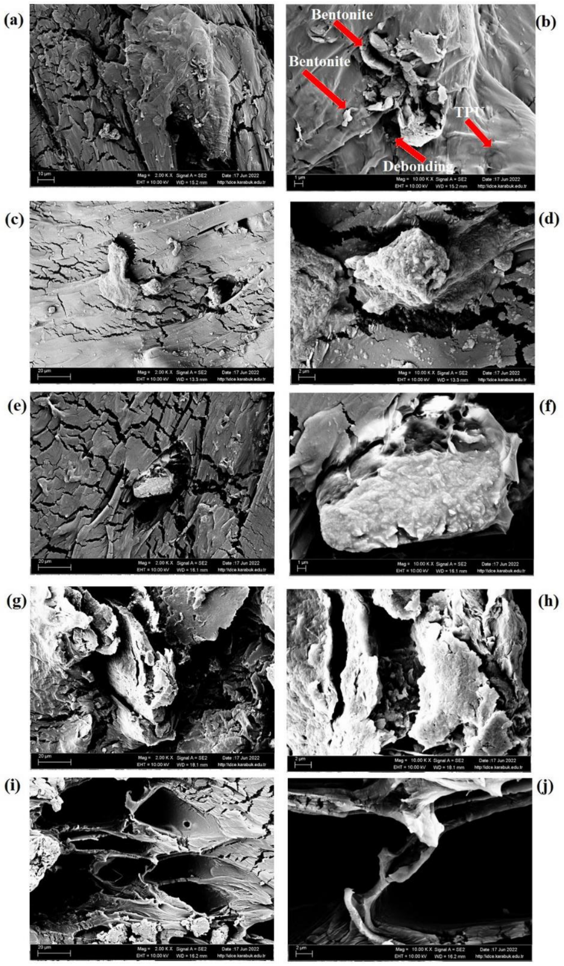

Figure 5. SEM micrographs of unaged TPU/BE composites (a-b), aged by UV in 2 weeks (c-d), aged by UV in 4 weeks (e-f), aged by UV+water in 2 weeks (g-h), aged by UV+water in 4 weeks (i-j).

In Figure 5, SEM micrographs of the samples of TPU/BE composites that were unaged and aged under various conditions are given. Due to the hydrophobic structure of TPU and the hydrophilic nature of bentonite, it was observed that there was no good adhesion in the composites and the bentonite particles were not fully embedded in the matrix. It was determined that ductile fracture behavior was observed in the TPU/BE composites that were not aged and applied. It was observed that the bentonite fragments became more free on the polymer surface with increasing UV times. On the other hand, it was observed that microcracks enlarged as water molecules entered and swelled between the polymer chain and bentonite particles in the samples that had been treated with UV+water aging (48).

SEM micrographs of bentonite-containing composite samples treated with silane surface modification are shown in Figure 6. Due to the hydrophobic nature of the silane, it was observed that the surface adhesion between the polymer matrix and the bentonite was better. According to the results obtained, it was observed that as the UV time increased, some micro-voids were formed and the adhesion of bentonite particles to the polymer matrix decreased in the sample that was UV applied for 4 weeks. It was observed that the ductile fracture behavior increased in the samples treated with UV+water.

CONCLUSION

Mechanical, thermal and morphological properties of pristine TPU and TPU based composites containing without and with surface modification (epoxysilaned) bentonite were investigated under the different artificial aging process. As a results;

1-) Hydrophobicity of bentonite increased after applying the silanization process and it is proven with ATR/FTIR.

2-) Considering the TGA of pristine TPU, it was observed that the thermal resistances of the samples that were treated with UV and UV+water artificial aging processes for 4 weeks were lower. It was observed that the thermal resistance of the 2-week-olds increased compared to pristi-

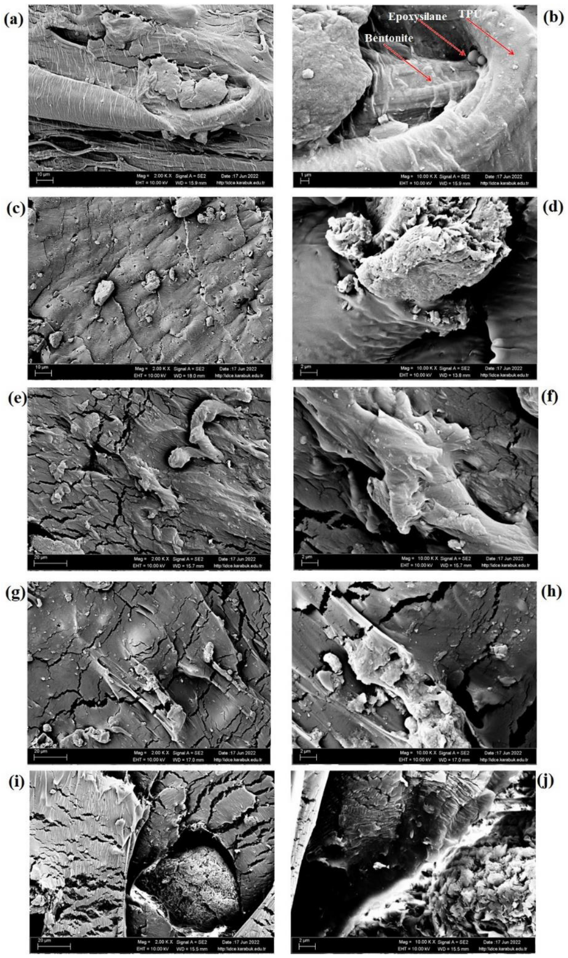

Figure 6. SEM micrographs of unaged TPU/ES-BE composites (a-b), aged by UV in 2 weeks (c-d), aged by UV in 4 weeks (e-f), aged by UV+water in 2 weeks (g-h), aged by UV+water in 4 weeks (i-j).

ne TPU. In the second step of thermal degradation with the addition of bentonite, it was observed that the thermal resistance of the sample, which was only UV applied for 2 weeks, was slightly higher than the other samples. In the second step of thermal decomposition with the addition of modified bentonite, the thermal resistances of pristine TPU and samples that were aged for 4 weeks with UV and UV+water were almost close to each other.

3-) UV exposure time affect the mechanical properties of composites. UV+water causes the cross-linking, so it causes the increase the Young's modulus and tensile strenght of the composites.

4-) In the FESEM of pristine TPU, the surface got roughness after artificial aging process and also it cause some voids. With the addition of bentonite, there was a big voids between the TPU and bentonite because of the incompability of surface. FESEM of TPU containing the modified bentonite composites, the voids got smaller and the micro-voids were occurred with increasing UV exposure time.

ACKNOWLEDGEMENTS

This work was supported by Research Fund of the Karabuk University. Project Number: KBÜBAP-22-DS-103. I would like to thank Karabuk University BAP Coordinator for their financial support.

CONFLICT OF INTEREST

There is not any conflict of interest or common interest with an institution/organization or a person that may affect the review process of the paper.

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