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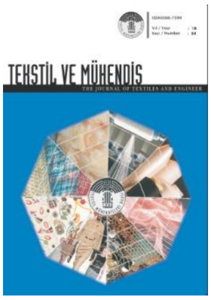

Full Factorial Experimental Design for Water Absorption and Wettability of Jute Fabric Reinforced Acrylated Epoxidized Soybean Oil/Epoxy Biocomposites

Jüt Kumaş Takviyeli Akrilatlı Epoksitlenmiş Soya Fasulyesi Yağı / Epoksi Biyokompozitlerin Su Absorpsiyon ve Islanılabilirliğine Yönelik Tam Faktöriyel Deneysel Tasarım

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FULL FACTORIAL EXPERIMENTAL DESIGN FOR WATER ABSORPTION AND WETTABILITY OF JUTE FABRIC REINFORCED ACRYLATED EPOXIDIZED SOYBEAN OIL/EPOXY BIOCOMPOSITES

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ABSTRACT: Today, composites, which offer advantages for many application areas, cannot be disposed of in an environmentally friendly way when their lifetime is over. This encourages the use of biodegradable resin systems alongside natural fiber reinforcement. In this study, jute fabric reinforced composites are manufactured via vacuum infusion technique using both bio-resin (acrylated epoxidized soybean oil (AESO)) and epoxy resin. The effects of bio-resin ratio (0-100%) and curing temperature (20, 90, and 120°C) on the water absorption and wettability properties of the composites are investigated by experimental and statistical analyses. Moreover, the obtained results are supported by differential scanning calorimetry analysis (DSC) and scanning electron microscopy (SEM) images. Water absorption and water contact angles of the composites are measured and a full factorial design is adapted to the test results to investigate the effects of bio-resin ratio, curing temperature as well as their interactions. Experimental results show that the increase in the bio-resin ratio increases the water intake, which causes an increase in the water absorption of the samples and a decrease in the water contact angle values. On the other hand, the heat treatment applied during curing helps to limit water absorption in samples, especially when the bio-resin content is above 50%, and this result is supported by higher water contact angle values. In terms of analysis of variance (ANOVA), both variables as well as the combined effects of these variables are found statistically significant in defining the behavior of composites against water.

Keywords: Water contact angle, water absorption, bio-resin, acrylated epoxidized soybean oil, biocomposite, curing temperature, full factorial design

JÜT KUMAŞ TAKVİYELİ AKRİLATLI EPOKSİTLENMİŞ SOYA FASULYESİ YAĞI / EPOKSİ BİYOKOMPOZİTLERİN SU ABSORPSİYON VE ISLANILABİLİRLİĞİNE YÖNELİK TAM FAKTÖRİYEL DENEYSEL TASARIM

ÖZET: Günümüzde birçok uygulama alanı için avantajlar sunan kompozitler, ömürleri dolduğunda çevreye duyarlı bir şekilde bertaraf edilememektedir. Bu durum, doğal elyaf takviyesinin yanı sıra biyolojik olarak parçalanabilir reçine sistemlerinin kullanımını da teşvik etmektedir. Bu çalışmada jüt kumaş takviyeli kompozitler hem biyo-reçine (akrilatlı epoksitlenmiş soya fasulyesi yağı) hem de epoksi reçine kullanılarak vakum infüzyon tekniği ile üretilmiştir. Biyo-reçine oranı (% 0-100) ve kürleme sıcaklığının (20, 90 ve 120°C) kompozitlerin su absorpsiyon ve ıslanabilirlik özelliklerine etkileri deneysel ve istatistiksel analizlerle araştırılmıştır. Ayrıca, elde edilen sonuçlar diferansiyel taramalı kalorimetri analizi ile taramalı elektron mikroskobu görüntüleri ile desteklenmiştir. Kompozitlerin su absorpsiyon ve su temas açıları ölçülmüş ve biyo-reçine oranı, kürleme sıcaklığı ve bunların ikili etkileşimlerinin tesirini araştırmak için test sonuçlarına tam faktöriyel deneysel tasarım uygulanmıştır. Deneysel sonuçlar, biyo-reçine oranındaki artışın su alımını arttırdığını, bunun da numunelerin su emiliminde artışa ve su temas açısı değerlerinde düşüşe neden olduğunu göstermektedir. Öte yandan, kürleme sırasında uygulanan ısıl işlemin, özellikle biyo-reçine içeriği % 50'nin üzerinde olan numunelerde su emilimini sınırlamaya yardımcı olduğu görülmüş ve bu sonuç, daha yüksek ölçülen su temas açıları ile desteklenmiştir. Varyans analizi açısından, kompozitlerin suya karşı davranışını tanımlamada hem değişkenler hem de bu değişkenlerin ikili etkileri istatistiksel olarak anlamlı bulunmuştur.

Anahtar Kelimeler: su temas açısı, su absorpsiyonu, biyo-reçine, akrilatlanmış epoksitlenmiş soya fasulyesi yağı, biyokompozit, kürleme sıcaklığı, tam faktöriyel tasarım

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1. INTRODUCTION

Today, the competitive market and ecological awareness, which is gaining momentum, bring the search for new materials that can fulfill the functions of existing materials and are compatible with the environment [1]. The biocomposites are composite materials in which at least one of the reinforcement or matrix materials is obtained from renewable resources [2]. On the other hand, green composite terminology is used for composites produced from both natural fibers and biodegradable resins [3]. In terms of reinforcement material, plant fibers serve as sustainable options. Among them, jute fiber is one of the most preferred natural reinforcement materials for composites with its biodegradability, high specific strength, commercially availability and low cost [4]. When the environmental footprints of the components that make up the composite materials are compared, it is seen that the use of petroleum-based matrix polymers rather than synthetic reinforcement fibers increases the footprint more [5]. On the other hand, due to the sustainability problem in the petrochemical industry and the demand for resources that are less harmful to the environment, the need for industrial raw material resources has increased, and vegetable oils have come to the fore as an alternative to petroleum-based resin derivatives [6].

Triglyceride-based vegetable oils are preferred in composite materials because they are environmentally friendly, have a relatively low cost and are easy to find [7]. Soybean oil, which has an important share in vegetable oils, is a preferred derivative in the form of acrylated epoxidized soybean oil (AESO) and offers good mechanical and thermal properties [8]. Due to their long aliphatic chains and a low degree of unsaturation, AESO resins have difficulties in production thus generally used in a mixture of several resin systems such as styrene and epoxy to improve mostly the damping properties of the composites [9, 10].

The water absorption feature of natural fiber reinforced biocomposites negatively affects the mechanical properties of composite structures, while reducing their dimensional stability. This may be caused by the use of natural fibers such as jute, which is hydrophilic in nature, as reinforcement material, or it may occur from undesired air gaps formed during composite production. As a result, this undesirable moisture absorption feature limits the end-use areas of the produced bio-composites in the outdoor applications. This problem needs to be solved in order to expand the industrial usage areas of bio-composites [11]. Not only the use of natural fiber reinforcement and production process but also using a bio-resin like AESO also increase the water absorption capacity of the composite. AESO is hydrophilic due to the polar hydroxy and epoxy groups in its molecular chain structure, which causes a high water absorption rate [5, 12]. Thus, this can also be assumed as one of the primary reasons of mechanical performance drop for bio-composites. To overcome these problems, studies generally focus on improving the fiber-matrix interface by applying chemical finishing to the natural fibers or modifiying the matrix systems with appropriate chemical subsitutes. However, the current study focuses on the production and curing temperatures of jute fabric reinforced AESO-epoxy matrix resin bio-composites and reveals the effect of this thermal effect on the moisture absorption and water contact angle properties of the composites, both by itself and in combination with the increased bio-resin ratio.

Since most of the studies focus on mechanical properties, there are only a few studies that have a partial contribution to the water absorption and wettability properties of these AESO based natural fiber-reinforced biocomposites. For instance, Liu et al. (2018) studied the effect of different reactive diluents used for AESO and their effects on the water absorption properties of hemp reinforced biocomposites [13]. Moreover, Kocaman et al. (2017) focused on the effect of the varying surface treatments applied for coconut wastes used as reinforcement materials on the water absorption properties of AESO based biocomposites [14]. Seabra et al. (2020) studied the recyclability of jute fabric reinforced AESO based biocomposites and its effect on water absorption characteristics [5]. However, in the literature, which generally focuses on the mechanical properties of biocomposites, there is no study examining the effect of curing temperature and increasing AESO ratio in hybrid epoxy composites on wettability and water absorbency, in details.

For this purpose, this study is conducted to examine the wettability and water absorbency properties of the composite structures with increased bio-resin ratio (0-100% wt.) and different curing temperatures (20, 90, 120°C) in 4-plied jute fabric reinforced AESO-epoxy based composites produced by the vacuum infusion method. In this context, a full factorial experimental design is carried out within the scope of the study and the individual and dual effects of both parameters are discussed.

2. MATERIALS AND METHODS

2.1. Materials

In this study, while jute plain woven fabric (Table 1) is used as reinforcement material, epoxy (F-1564, Fibermak, d=1.15gcm^{-3}, μ=1200-1400mPa.s) and acrylated epoxidized soybean oil (AESO, Sigma Aldrich, d=1.04gcm^{-3}, μ=18000-32000mPa.s) are used as the matrix materials. In addition, 25% by weight of hardener (F-3486, Fibermak, d=1.00 gcm^{-3}, μ=10-20mPa.s) is added to cure the resin system.

Table 1. Properties of jute fabric

Areal density	260 g/m^2
Warp yarn count	252 tex
Weft yarn count	252 tex
Count of cloth (epc x ppc)*	7 x 7
Thickness	0.72 mm

*epc: ends per cm, ppc: picks per cm

2.2.Method

2.2.1. Composite Production

The vacuum infusion technique is used for the production of composite samples. While it is preferred to use 4 layers of jute fabrics as reinforcement material in all samples, the composites are differentiated by changing the AESO:epoxy ratio (0:100, 30:70, 50:50,70:30, and 100:0 wt.) and the curing temperatures (20, 90, and 120°C). While the curing process at 20°C takes place for 48 hours without any heat treatment, the curing at 90°C is completed in 2 hours by heating the vacuum infusion table. For the curing process at 120°C, the composites cured at 90°C are exposed to additional heat in the oven for 2 hours. Production stages can be seen in Figure 1 and the details of the composites designed within the scope of the study are given in Table 2.

2.2.2. Water absorption test

Composite samples are first kept in the conditioning oven at 50°C for 24 hours. After that, it is kept in sealed plastic bags to cool down to room temperature and the dry weights (w_d) are weighed. Afterward, it is kept in distilled water at 20°C±2°C for 24 hours and at the end of the period, the excess water is removed with a tissue and the wet weight (w_w) is weighed. Equation 1 is used to calculate the water absorption values of the composite samples (ASTM D570-10). The average values of three samples tested for each sample group are given with error bars. Besides, swollen sample visuals are also introduced to bar graphs.

$$WA = \frac{w_w - w_d}{w_d} \, x \, 100 \qquad (1)$$

2.2.3. Water contact angle test

Water contact angle measurements are carried out with a KSV Attension Theta Lite device (working principle of sessile drop measurement) following the ASTM D7334-08 standard. The contact angle values are given as the average values of the right and left sides of the droplets and the graphs include droplet visuals along with error bars.

2.2.4. Differential scanning calorimeter

Differential scanning calorimetry analyzes of the composite materials produced within the scope of the study are carried out in the DSC 4000 (Perkin–Elmer, USA) device, and the samples are heated from -30 °C to 300 °C at a rate of 20 °C/min and then cooled to -30 °C at the same speed. This process is carried out in 2 cycles in order to remove the impurities in the material and the analyzes are made over the 2nd cycle.

Table 2. Composite designs

Sample codes	AESO:epoxy weight ratio	Curing temperature (°C)
0BIO	0:100	20
0BIO-90	0:100	90
0BIO-120	0:100	120
30BIO	30:70	20
30BIO-90	30:70	90
30BIO-120	30:70	120
50BIO	50:50	20
50BIO-90	50:50	90
50BIO-120	50:50	120
70BIO	70:30	20
70BIO-90	70:30	90
70BIO-120	70:30	120
100BIO	100:0	20
100BIO-90	100:0	90
100BIO-120	100:0	120

2.2.5. Scanning electron microscope (SEM) analysis

JSM 6060 LV (Jeol, Japan) scanning electron microscope was used to examine the morphologies of the composite samples.

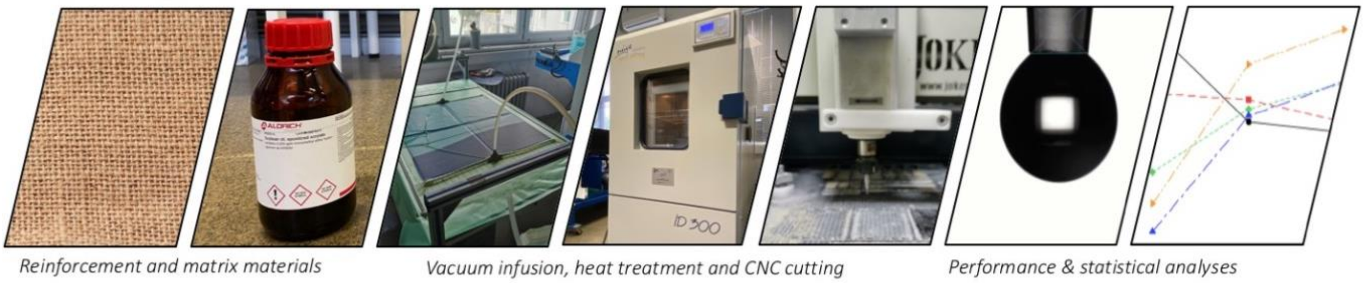


Figure 1. Production stages of bio-composites

2.2.6. Statistical analysis

The statistical analyses are conducted with Minitab 16 Software program. The full factorial experimental design layout is used to investigate the effect of the main factors (bio-resin content and curing temperature) and their interactions on the wettability and water absorption properties of composite structures. One factor with five levels and one factor with three levels are chosen to design the experimental study (Table 3). Each test is done three times for each combination of factors and levels and this leads to a total of 45 runs in the experimental design layout. ANOVA tables are utilized to examine the significance of the results. In ANOVA tables, the DF, Adj SS and Adj MS refer to the degree of freedom, the adjusted sum of squares and adjusted mean squares, respectively. The F value is the ratio of the MS (variable) to the MS (error), while the p-value is the area under the proper null-sampling distribution of F greater than the observed F-statistic. Since the statistical analysis is performed at a 95% confidence interval, it shows that the effect of variables with a p-value less than 0.05 on the outcome is significant [15, 16].

3. RESULTS AND DISCUSSION

3.1 Water Absorption

The water absorption rates of the composite samples and their visuals after the water absorption test are given in Figure 2.

When the results are examined, it is seen that the curing process reduces the water absorption values of the composite materials with varying bio-resin contents. It has been proven by previous studies that the crosslinks in the material increase as a result of the heat treatment of soybean oil resin and consequently the water intake decreases [17]. The obtained results also support this situation. Moreover, it can be easily seen that the increased bio-resin ratio increases the water absorption properties of the composite materials.

The ANOVA response table of water absorption test results is given in Table 4. From the p-values (0.000 <0.05), it is understood that both factors have a significant effect on the water absorption property of the composite material. When the Adj SS values are examined, it is seen that the change in the bio-resin ratio (Adj SS: 29948.3) makes a more significant difference in the water absorption property compared to the change in curing temperature (Adj SS: 897.3). This situation can also be seen from the main effect plots (Figure 3a). With the increasing bio-resin ratio, the increase in water absorption has increased with great acceleration. When compared with the curing effect, as seen in the main effects plot, the change in water absorption with increasing bio-resin ratio reaches 70%, while the change in water absorption value with curing effect is about 10%.

Table 3. Factors and levels used in full factorial experimental design.

Factors	Level 1	Level 2	Level 3	Level 4	Level 5
Bio-resin content (%)	0	30	50	70	100
Curing temperature (°C)	20	90	120		

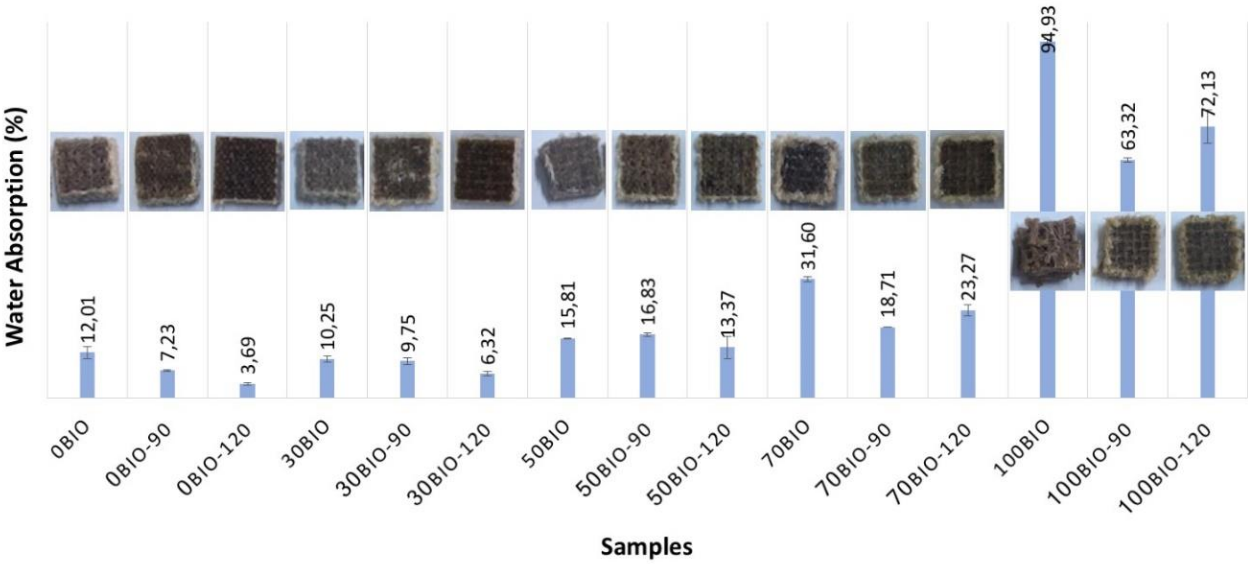
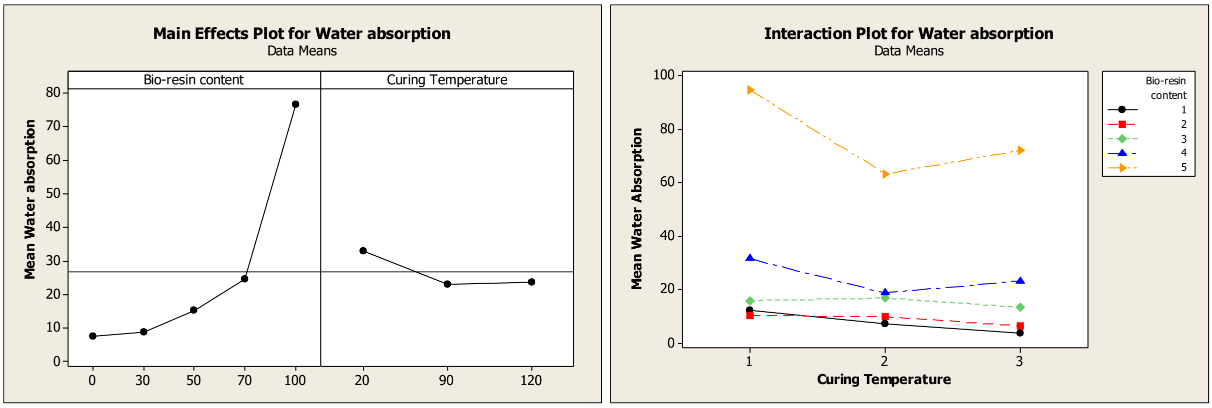


Figure 2. The water absorption test results

Table 4. Analysis of variance response table of water absorption test results.

Source	DF	Adj SS	Adj MS	F	P
Bio-resin content	4	29948.3	7487.1	2622.68	0.000
Curing temperature	2	897.3	448.7	157.17	0.000
Bio-resin content*Curing temperature	8	1106.8	138.3	48.46	0.000
Error	30	85.6	2.9		
Total	44				

Model summary: R-Sq = 99.73%   R-Sq(adj) = 99.61%



a **b**

Figure 3. (a) Main effects, **(b)** interaction plots for water absorption.

Considering the effect of the interaction of the factors on the water absorption values of the samples taken from the ANOVA table, it is seen that the Adj SS value of the bio-resin ratio * curing temperature interaction (1106.8) is higher than the Adj SS value of the curing temperature factor (897.3). This shows that the bilateral interaction is more effective on the water absorption of composite materials than the curing temperature. The lines being parallel to each other in the interaction plot shows that there is no intersection of these factors [18]. Although there seems to be parallelism in certain parts of the lines, it is possible to say that they are not exactly parallel to each other when examined carefully. The fact that the R-Sq (0.9973) and R-Sq (adj) (0.9961) values of the model are very close to 1 indicates the adequacy of the model.

3.2. Water contact angle test

The water contact angles of the composite samples and the droplet visuals obtained are given in Figure 4.

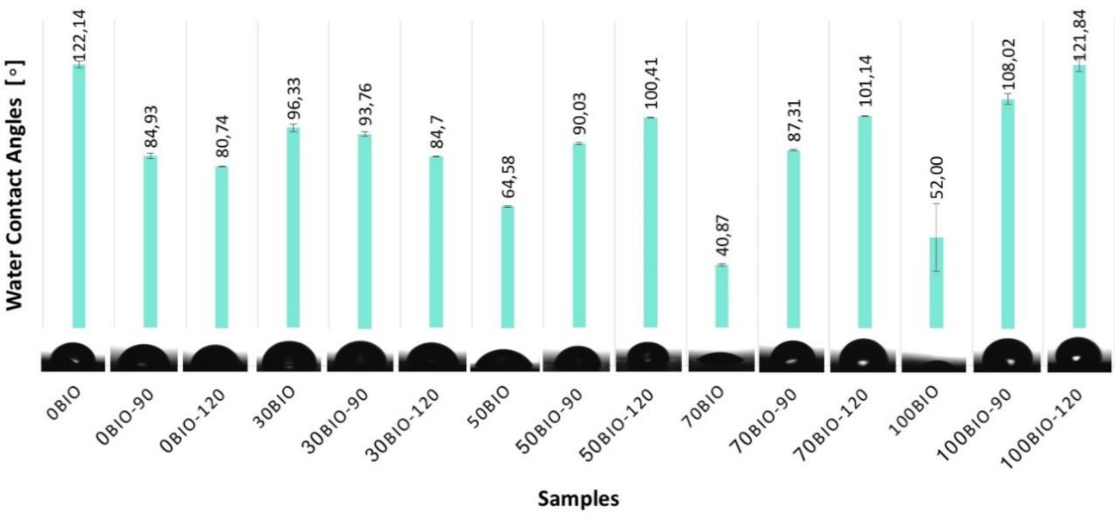


Figure 4. The water contact angle test results

When the effects of the curing process applied to composite materials with different bio-resin ratios on the wettability of the material are examined, it is seen that the water contact angle decreases with curing in the 0BIO and 30BIO samples, while curing processes applied to samples with 50% or more bio-resin results in an increment of the water contact angle and so the hydrophobicity of the sample. After heat treatment, the crosslinks in the AESO resin increase and this situation makes it difficult for the water droplets left on the material to introduce into the composite structure [17]. When the effect of the bio-resin ratio on the hydrophilicity of the composite material is examined without considering the curing process, it can be said that the water contact angle decreases considerably with the increasing bio-resin ratio (except 100BIO sample), in other words, the hydrophilicity of the material increases.

The ANOVA response table of water contact angle test results is given in Table 5. Firstly, when the p-values showing the statistical significance of the results are examined, it is seen that both factors (bio-resin content and curing temperature) and the interaction of these factors have a significant effect (p: 0.000<0.05) on the water contact angle measurement results of the materials. When the effect ratios of the selected two factors on the water contact angle test result are examined with Adj SS values, it is seen that, unlike the water absorption test results, the curing temperature (Adj SS: 4234.2) has a greater effect than the bio-resin ratio (Adj SS: 2273.4).

When the main effects plot (Figure 5a) is evaluated, it is seen that the water contact angle decreases with increasing bio-resin ratio up to 70BIO, but shows an increase in 100BIO sample, while the curing effect increases the water contact angle regularly. This explains the difference between the Adj SS values of these two factors. It is seen that the Adj SS value (15323.1) of the binary interaction is considerably higher than that of both factors. This indicates that the combination of increasing bio-resin ratio and increasing curing temperature has a very important effect on the wettability of the composite material. The fact that the lines in the interaction plot (Figure 5b) completely intersect also supports this result.

The fact that the R-Sq (0.9960) and R-Sq (adj) (0.9941) values of the model are very close to 1 indicates the adequacy of the model.

3.3 Differential scanning calorimeter (DSC) analysis

DSC analysis graphs of samples cured at different temperatures are given in Figure 6. When all three graphs are examined, it is seen that the glass transition temperatures (T_g) of the composite materials decrease with the increasing bio-resin ratio. This is expected given that the T_g of epoxy is between 48-52°C [19], and T_g of AESO is approximately -2 [20]. T_g value, which increases with the increase of cross-links, decreases with increasing AESO ratio, and this supports the increase in water absorption properties and the decrease in water contact angle values of the samples with high AESO ratio.

When the effect of different curing temperatures on the T_g values of the composite samples is examined, it is seen that there is no significant difference. In order to remove the impurities in the material and to obtain more accurate information about the material, analysis is carried out as 2 cycles. It is thought that the effect of curing is not seen in the second cycle, since the material is heated to 300°C in the first cycle of the DSC analysis.

3.4 Scanning electron microscopy analysis

SEM images of selected composite samples are given in Figure 7.

Table 5. Analysis of variance response table of water contact angle test results.

Source	DF	Adj SS	Adj MS	F	P
Bio-resin content	4	2273.4	568.4	192.51	0.000
Curing temperature	2	4234.2	2117.1	717.09	0.000
Bio-resin content*Curing temperature	8	15323.1	1915.4	648.77	0.000
Error	30	88.6	3.0		
Total	44				

Model summary: R-Sq = 99.60%   R-Sq(adj) = 99.41%

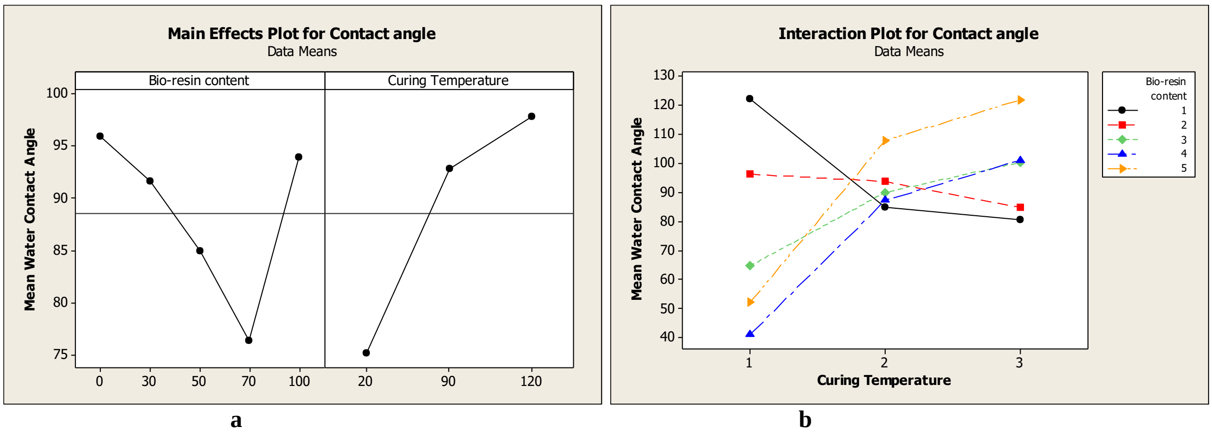


Figure 5. (a) Main effects, **(b)** interaction plots for water contact angle.

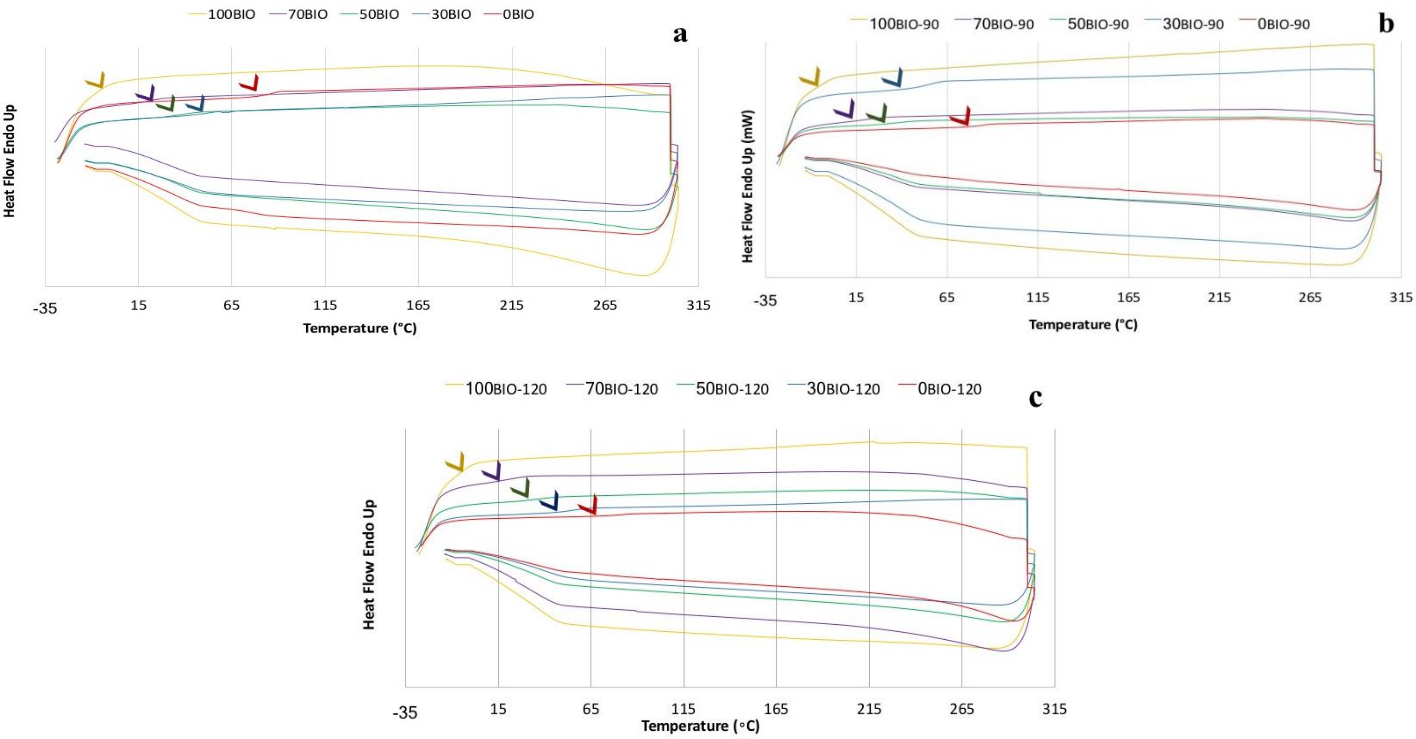


Figure 6. DSC curves of composite samples cured at **(a)** room temperature, **(b)** 90°C and **(c)** 120°C

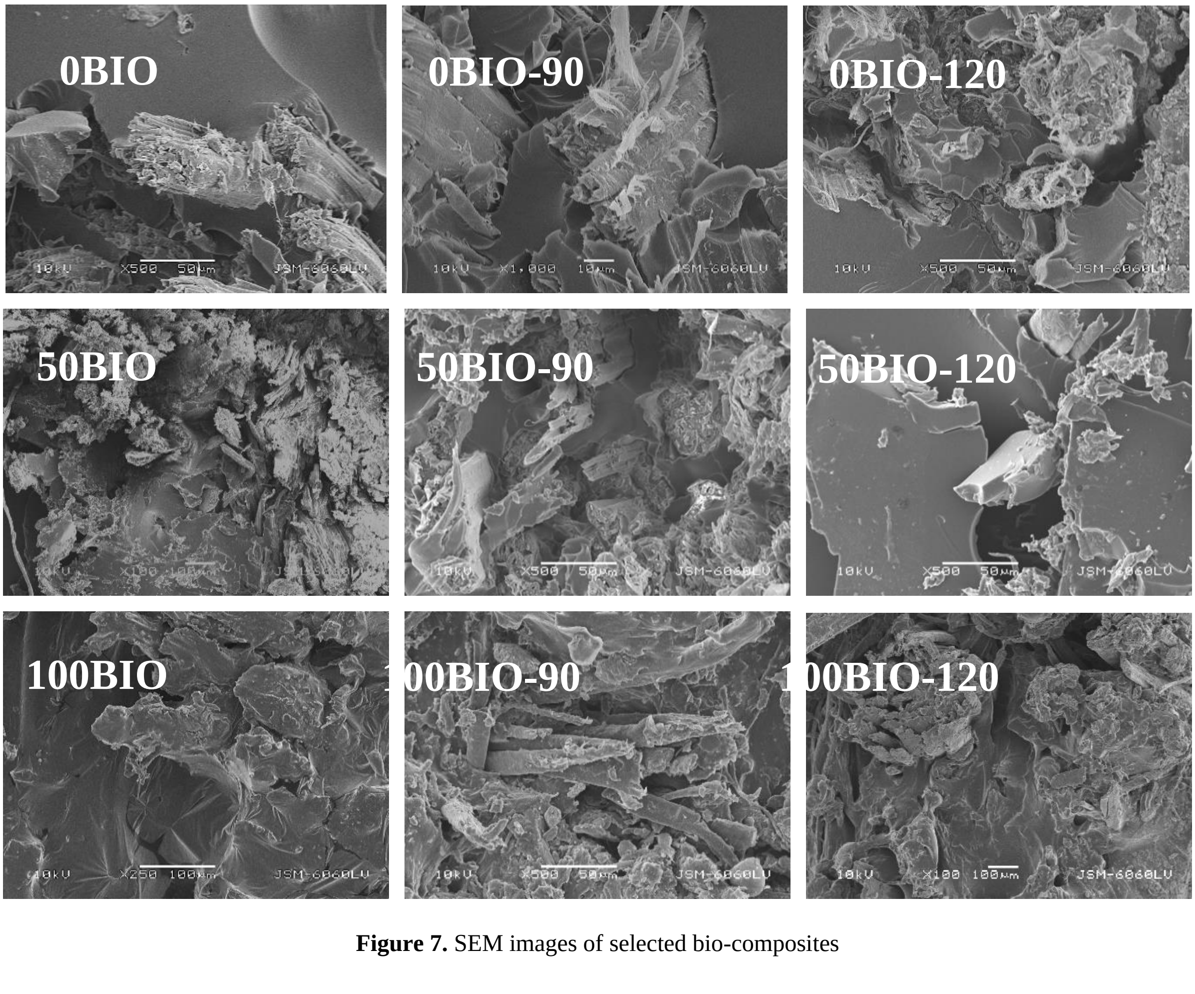


Figure 7. SEM images of selected bio-composites

When the SEM images are examined, it is observed that both the increased bioresin ratio and the applied heat treatment effect create a noticeable difference especially in the structure of the matrix phase. When the selected samples (0BIO, 50BIO and 100BIO) are examined, it is observed that the brittle structure of the matrix phase is lost with increasing bio-resin, while this loose matrix phase turns into a brittle form as in high epoxy percentages with increasing heat treatment.

4. CONCLUSION

In this study, jute woven fabric reinforced biocomposite materials are cured at three different temperatures using different ratios of AESO and epoxy resins. Using these two factors, a full factorial experimental design is created and the effects of these factors on the water absorption and wettability properties of the composite materials are statistically analyzed. The results obtained show that both factors have a statistically significant effect on water absorption and wettability properties. While the most important factor on water absorption is found to be the ratio of bio-resin, it has been observed that the dual combination of these two factors is the most effective on the water contact angle. Regardless of the curing effect, it is seen that while the water absorption property increases by approximately 700% with the increase in the bioresin ratio, the water contact angle decreases by 50%. It is observed that the water contact angle value increase by 100% with increasing curing temperature in samples with a bioresin ratio of 50% or more. Moreover, DSC analyzes of the obtained samples are done and fiber-matrix interfaces are examined with SEM images. As a result of the DSC analysis, it is determined that the T_g values of the composite materials decreased with the increasing bio-resin ratio, while the brittle structure formation in the matrix material is observed with the increasing curing temperature from the SEM images. It has been seen that the high hydrophobic property expected from the material can be achieved with increasing curing temperature and decreasing bio-resin ratio in hybrid biocomposite structures.

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