



Examining the breaking and bending behaviors of the polymethylmethacrylate composites reinforced with hazelnut shell powder

Fındıkkabuğu tozu takviyeli/polimetilmetakrilat kompozitlerin kırılma ve eğilme davranışlarının incelenmesi

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Eser Bilgisi/Article Info

Araştırma makalesi/Research article

DOI: 10.17474/artvinofd.532204

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Geliş tarihi / Received

25.02.2019

Düzeltilme tarihi / Received in revised form

17.04.2019

Elektronik erişim / Online available

26.06.2019

Keywords:

Composite

Hazelnut shell powder

Mechanical properties

PMMA

Fracture toughness

Anahtar kelimeler:

Kompozit

Fındıkkabuğu tozu

Mekanik özellikler

PMMA

Kırılma tokluğu

Abstract

The objective of the present study is to reveal the toughening effect of the hazelnut shell on the brittle matrices by using the bending and breaking methods. The polymethylmethacrylate composites reinforced with hazelnut shell powder with 50 μ particle size at the weight concentrations of 5%, 10%, 15%, and 20% were prepared. The polymer beam samples were exposed to heat cure procedure and the initial notches were created at $a/W=0.1, 0.2, 0.3, 0.4,$ and 0.5 . The Mode I breaking behaviors of the notched at single edge and non-notched composite samples were examined by using three-point bending test. The critical stress intensity factor (K_{IC}), J-integral, initial notch depth, and compliance methods were used in calculations. Besides them, the bending module and bending stress values were calculated. The microstructures of HN/PMMA composites were determined by using XRD (x-ray diffractometer), FTIR (Fourier Transform Infrared Spektrofotometre), and SEM (scanning electron microscope) analyses. According to the results obtained, it was determined that the bending strength, elasticity module, and fracture toughness values increased at 10% additive ratio for the HN/PMMA (polymethylmethacrylate) composites.

Özet

Çalışmanın amacı, gevrek matrislerde fındıkkabuğunun toklaştırma etkisini eğilme ve farklı kırılma yöntemleri ile ortaya koymaktır. 50 μ partikül boyutuna sahip %5, %10, %15 ve %20 ağırlık oranlarında fındıkkabuğu tozu takviyeli /Polimetilmetakrilat kompozitler üretilmiştir. Üretilen polimer kirış numunelere, ısıl kür işleminden sonra, $a/W=0.1, 0.2, 0.3, 0.4, 0.5$ başlangıç çentikleri açıldı. Çentiksiz ve tek kenardan çentik açılmış kompozit numunelerin mode I kırılma davranışları üç nokta eğme testi uygulanarak ortaya konuldu. Kritik Gerilme Şiddet Faktörü (K_{IC}) J- integral, Başlangıç Çentik Derinliği ve Komplians metotları ile hesaplandı. Bununla birlikte eğilme modülü ve eğilme gerilmeleri de belirlendi. HN/PMMA kompozitlerin mikro yapısı XRD, FTIR ve SEM çalışmaları ile ortaya konuldu. Çalışmanın sonuçlarına göre, fındıkkabuğu tozu / PMMA kompozitlere eklenen tozların %10 katkı oranında eğilme kuvveti, eğilme modülü ve kırılma tokluğunu arttırdığı belirlenmiştir.

INTRODUCTION

The use of sustainable resources gradually increases day-by-day because of the increase in environmental awareness Asadi et al. (2018); (Spanic et al. 2010). From this aspect, in the previous century, the strategies aiming the use of wastes as raw material were implemented in many industries (Sozen et al. 2017).

Nowadays, thanks to the increasing interest in biomaterial studies, the composite materials that damage the nature at a lower level and can be degraded in a short period were developed. Some of the additive and reinforcement materials used in these studies were the natural materials and natural wastes. Some of these natural materials are short sisal (Agave sisalana) fibers

Luyt et al. (2005), jute (Corchorus capsularis, Corchorus olitorius) fibers Gassan et al. (1997), wheat and rye shell Bledzki et al. (2010), and tree powders (Guo et al. 2013).

In our country, the meat of hazelnut grown generally in the Black Sea region is utilized and the shell is generally used as fuel. Although it varies between the years, the average annual production of hazelnut is 592,000 tons and the shell constitute approx. 48% (Ayfer 1986: Commodity Exchange Data 2009). One of the general objectives of this study is to find an area of industrial use for this material, which has approx. 300 000 tons of annual supply, rather than using as fuel.

As in all the other materials, the composite materials may also be subject to various damages under the working

conditions. The fracture damage is one of the most important damages seen in the composite materials. For this reason, many researchers carried out studies on the composite materials and aimed to reveal the breaking behavior of the composites. From this aspect, the researchers investigated the effects of organic and inorganic fillers (sand, tree powder) on the mechanical and breaking behaviors of the composites (Davalos et al. 1998; Arıkan et al. 2004; Samancı 2012; Vipulanandan et al. 1989).

There are very few studies carried out on the use of hazelnut shell powder in the composites and the mechanical properties of these composites. In one of these studies, Balart et al. (2016) investigated the effects of plasticizer (ELO) on the hazelnut shell powder/PLA composites and reported that the plasticizer decreased the fracture sensitivity of the composite and increased the thermal stability.

In the present study, the hazelnut shell powder that is a waste product was used as filler material in the composites with polymethylmethacrylate matrix. The three-point bending tests of the notched and non-notched samples were performed in accordance with ASTM E-399 standards, whereas the stress intensity values were determined by using J-integral, initial notch depth, and compliance methods. The results obtained from the present study showed that the hazelnut shell powder added to PMMA increased the bending strength and elasticity module and also the fracture toughness at the highest level at 10% concentration.

MATERIAL AND METHOD

Preparation of the Samples

The hazelnut shell obtained from the hazelnut cracking factories were ground to powder form in local flour mills. As a result of the preliminary analyses, it was determined that the optimum results were obtained from the PMMA/HNSP composites containing hazelnut shell powder at the particle size of $0.50\mu\text{m}$, thus the hazelnut shell powder with the maximum particle size of $50\mu\text{m}$ was used in the present study.

The matrix material used in preparing the HNSP/PMMA composites was procured from the Turkey distributor of

Otto Bock (Germany). In order to harden the polymethylmethacrylate matrix material, the peroxide-based chemical material was also obtained from the same company. Some of the specifications of standard PMMA material are presented in Table 1 (Xiaofei et al. 2014; Marshall et al. 1974).

Table 1. Some of the mechanical properties of standard PMMA

Elong at Break(%)	0.5-5
Hardness(M)	93
Impact resistance (kJ/m^2)	11
Poisson Ratio	0.35-0.40
Elas Module (MPa)	3000
Tens strenght (MPa)	40-70
Fracture toughness ($\text{MN/m}^{3/2}$)	0.7-1

The casting molds were prepared by using PTFE material in order to obtain the experimental PMMA/HNSP specimens. The composites were prepared by mixing hazelnut shell powder into PMMA matrix at predetermined concentrations (5, 10, 15, and 20%). 2% peroxide hardener was added in order for the composites to harden. The prepared liquid mixture was poured into the molds and the unprocessed test samples were obtained. Then, for the final cure, the samples were kept in the oven for 24 hours at 80°C . The samples were cut to $70\times 14\times 7\text{mm}$ size by using an automated device (Figure 1). The standard notches with a/w ratios of 0.1, 0.2, 0.3, 0.4, and 0.5 were created on the samples.

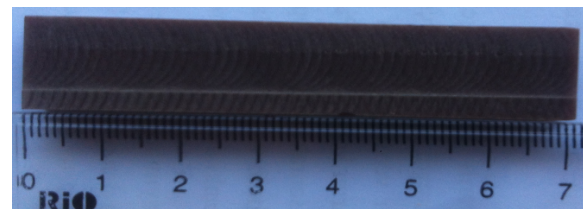


Figure 1. Casted and processed composite sample without notch for 3-Point Bending Test

Test Methods

Characterization

The FTIR analyses of the composites were performed by using Perkin Elmer Spectrum1400 spectrometer at $4000\text{-}650\text{ cm}^{-1}$ scanning rate and at room temperature. The test was performed with 10 repeats at 2 cm^{-1} resolution.

X-ray diffraction test was performed by using advanced Diffractometer (Europe 600 XRD) with copper radiation. Cu Ka was run at 40 kV and 30 mA. The scanning rate was

set to 10-30, the feeding rate to 0.02, and the scanning rate to 0.4 min⁻¹.

The scanning electron microscopy image was obtained by using JEOL Ltd. JSM-5910 after plating with gold. The SEM analysis was performed by examining the fractured surfaces of HNRP/PMMA composites.

Three-point bending test (TPB)

The bending strength (σ_f), elasticity module (E_f), and fracture toughness (K_{IC}) analyses were performed by using three-point bending method with rectangular bars with 70x14x7mm dimensions. The gap at the notched edge was measured by using clip-gage (SHIMADZU P701805). The bending test was performed by using 1.0 mm/min. feeding rate in SHIMADZU EHF-LV020K2-020 device (Figure 2). σ_f and E_f values were calculated by using the equations below (Atkins et al., 1988):

$$\sigma_f = \frac{3PmS}{2WD^2} \quad (1)$$

$$E_f = \frac{PmS^3}{4WDe} \quad (2)$$

Here, Pm refers to the load at the moment of breaking, S to the distance between bearings, D to the sample thickness, e to the deflection in the sample, and W to the sample width.



Figure 2. Fracture test setup

J-integral method

The J-integral method is a technic used for calculating the stress-energy oscillation ratio or the energy per unit of fractured surface area and it is calculated by using Equation 3 (Cherepanov 1974). TPB test was applied to

both notched and non-notched samples and the areas below the load-displacement curve were determined by using Matlab software. The values were put to Equation 3 and the critical j-integral value was calculated.

$$J_{IC} = \frac{2(A_t - A_u)}{b(W-a)} \quad (3)$$

Here, (A_t) and (A_u) are the areas below the load-displacement curve for notched and non-notched samples, respectively. Moreover, a refers to the initial notch depth, and W and b to the dimensions of samples in Figure1. By using the J_{IC} data obtained from Equation 3, the K_{IC} values were calculated (Ted et al. 2005).

$$K_{IC}^2 = \left(\frac{E}{1-\nu^2} \right) J_{IC} \quad (4)$$

Here, ν refers to the Poisson ratio and E to the elasticity module.

Initial notch method

This method was developed by Tada et al. (2000) in order to calculate the linear elastic breaking mechanic stress intensity under TPB test conditions. This method is also known as the initial notch method and Mode I stress intensity is calculated by using Equation 5.

$$K_{IC} = \left(\frac{3PS}{2W^{3/2}} \right) f \left(\frac{a}{W} \right) \quad (5)$$

Here, P refers to the maximum load for the expansion of notch, S to the distance between bearings, W to the sample height, b to the sample width, a to the notch depth, a/W to the notch ratio, and f(a/W) to the correction constant for the initial depth of sample and is calculated by using Equation 6.

$$f \frac{a}{W} = \frac{1}{\sqrt{\pi}} \frac{1,99-A(1-A)(2,15-3,93A+2,7A^2)}{(1+2A) \left(\sqrt[3]{1-A} \right)} \quad (6)$$

A=a/W is the initial notch ratio.

Compliance method

It is a method that is suitable for calculating the critical stress intensity for the brittle materials (Underwood 1986). In this method, the load-CMOD diagrams of the samples with different notch depths were used (Figure 7)

and the compliance values (C_1, C_2, \dots, C_n) were calculated by using Equation 7.

$$C = \frac{dCMOD}{dP} \quad (7)$$

$C=f(a/W)$ function, which is the compliance notch-depth ratio comparison, was obtained by using the curve equation in Figure 8. The derivation of $C=f(a/W)$ function to (a/W) was put into Equation 7 and the (K_{IC}) values of the samples were calculated by using Equation 8.

$$K_{IC} = \frac{E}{1-\nu^2} \frac{P}{2b} \frac{dC}{d(a/W)} \quad (8)$$

E refers to the elasticity modulus of the samples, P to the maximum load applied, ν to the Poisson ratio, and $\left(\frac{dC}{d(a/W)}\right)$ to the comparison curve of compliance notch-depth ratio.

RESULTS AND DISCUSSION

FTIR Analyses

Given the FTIR analysis presented in Figure 3, it is thought that the peak at 750 cm^{-1} arose from the vibration of the C-C bond (Sims 2002). The peak at 1057 cm^{-1} occurs because of the C-H vibration in the aromatic ring and C-O vibration in primary alcohols (Kumar 2009). The peak at 1433 cm^{-1} confirms the presence of ester bridges between the ring structures in hemicellulose or the groups such as ester or phenol within the structure of cellulose. The peak at 1632 cm^{-1} represents the C=O vibration within the structure of hemicellulose and confirms the presence of ester, ketone or aliphatic acid groups within the structure (Courtney et al. 2010). The peak at 2946 cm^{-1} represents the CH vibration band and is dependent on the alkyl groups. The peak observed at 3338 cm^{-1} suggests the presence of O-H groups. This peak is believed to arise from the moisture or alcohol or phenol groups that the hazelnut shell contains (Açikalin et al. 2010; Özveren et al. 2012). Given the FTIR analysis of the composite, the presence of hazelnut shell within the structure of composite can be easily confirmed. In the presence of hazelnut shell, the slight shifts occurred in some of the peaks of PMMA. This suggests that there is a weak relationship between two materials.

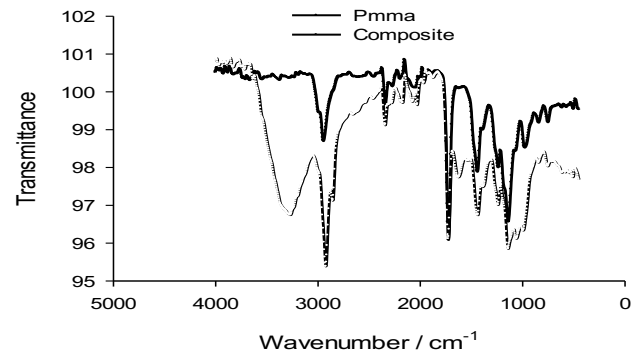


Figure 3. FTIR Image of 10% HNSP-PMMA composite

X-Ray Diffraction Analysis of Hazelnut Shell Dust

The X-Ray diffraction patterns of the typical crystal cage of the cellulose within the structure of hazelnut shell are presented in Figure 4. Two theta values of the main patterns belonging to the cellulose fiber are 16 and 21.8° , respectively, and these patterns correspond to the planes (101) and (002). The peak (002) is the peak of the largest crystal of cellulose. The crystallinity index (CrI) of the hazelnut shell powder was determined by using the Segal empirical method (Mwaikambo et al. 2002; Roncero et al. 2005). In some of the previous studies, the same method was used for measuring the crystallinity indices of various fibers such as sisal, linen, cannabis, and kenaf (Rong et al. 2001).

$$CrI = \left(\frac{I_{002} - I_{am}}{I_{002}} \right) \times 100 \quad (9)$$

Using Figure 9, the I_{002} and I_{am} values were determined and it was found by using Equation 1 that the crystallinity index of the hazelnut shell powder with a particle size of $50 \mu\text{m}$ was 31% (Büyükkaya 2017).

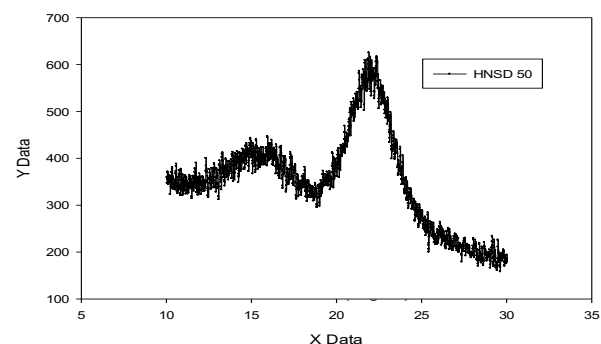


Figure 4. XRD pattern of 0-50 hazelnut shell flour

Bending strength and modules

The change of bending strength and elasticity module values of composites by the additive concentration is presented in Figure 5. In parallel with the increase in the concentration of additive, the bending strength values decreased and the decrease was found to be 14%, 5%, 8%, and 10% for the additive concentrations of 5%, 10%, 15%, and 20%, respectively. The increases in elasticity module were found to be 29%, 64%, 49.60%, and 57%, respectively. Accordingly, it was determined that the elasticity module values increased in the composites containing 10% additive but both of the module and bending strength values started decreasing after this point. Bhaskar et al. (2011) reported that the bending strength of the wood-plastic composites they examined has decreased and the elasticity module has increased as the amount of additive increased.

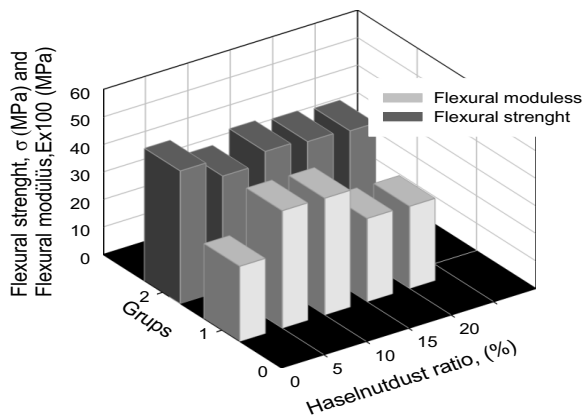


Figure 5. Flexural stress and modules versus percentage of 10% HNSP 0-50 reinforcement

Critical Stress Intensity Factor

The stress intensity factor (K) used in fracture mechanics is a measure used in determining the stress area around the crack and it depends on the geometrical size of the material, load type, and location and direction of crack. The fracture toughness is a characteristic of material and is defined with critical stress intensity factor (K_{IC}). When the stress intensity factor (K_{IC}) is reached, the irregular crack occurs. K_{IC} values are used for determining the size of the crack when a specific level of stress is applied to the material or calculating the critical stress value when a crack occurs on a material (George et al. 2007).

The researchers have developed different methods for calculating the stress intensity (K_{IC}). In the present study, the J-Integral, Initial Notch and Compliance methods were used for calculating the critical stress intensity values and these values are presented in Figure 9.

J-Integral Method

Load-displacement diagrams of the notched and non-notched samples are presented in Figure 7. The areas under both curves were determined by using Matlab software. The values obtained were put into Equation 3 and J_{IC} values were calculated. J_{IC} values were used in Equation 4 and the obtained K_{IC} values are presented in Figure 10. When compared to the pure matrix, the K_{IC} values of composites containing 5%, 10%, 15%, and 20% additive were higher by 8%, 49%, 26%, and 6%, respectively. In their study, Zarges et al. (2017) determined the fracture toughness of propylene/glass fiber and cellulosic fiber composites by using the J-integral method. They reported that cellulosic fibers had higher fracture toughness values than glass fibers did.

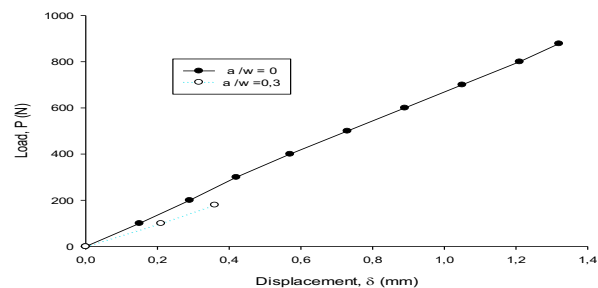


Figure 6. Load-Displacement diagrams of the notched and non-notched polymer composite beams

Initial Notch Depth Method

The critical stress intensity factor (K_{IC}) of the composite material was calculated by using Equation 5 according to the initial notch depth method and presented in Figure 9. When compared to the critical stress intensity factor of the pure matrix, it was determined that the K_{IC} values of the polymer composite containing 5%, 10%, 15%, and 20% additive were found to be higher by 15%, 51%, 32%, and 17%. The data showed that the highest stress intensity factor was obtained with 10% additive concentration. Similar results were also obtained in some of the previous studies. In one of those studies, Kim et al. (2008) reported that 1.5% nano-clay additive increased

the fracture toughness of the epoxy composites by 46% and 3% carbon black additive increased in by 23%.

Compliance Method

In the present study, the compliance (C) values were calculated by using the slope of the load-CMOD curve (Figure 7). The diagram of C-2a function was prepared by using the obtained values (Figure 8). K_{IC} values were calculated by using the derivation of obtained function in Equation 8 and they are presented in Figure 9.

When compared to the K_{IC} values of pure matrix, the addition at 5, 10, 15, and 20% concentrations increased the fracture toughness of tested composite samples by 16%, 55% 36%, and 22%, respectively. Rather than the standard calibration, the experimental calibration was used in the present study.

Creel et al. (2008) used two different calibrations in compliance method. One of them is ASTM standard calibration values and the second one is the experimentally obtained calibration data. The authors reported that the values of standard calibration applied to the anisotropic biological materials such as bones are different from the experimental calibration values.

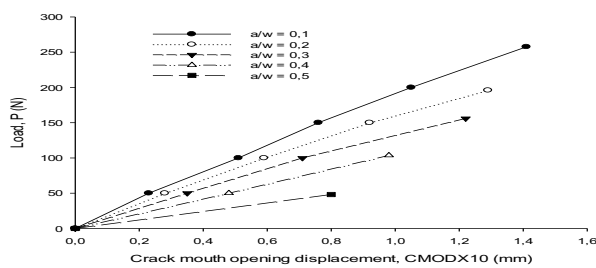


Figure 7. Load-CMOD variations of 10% HNSP-reinforced composites with various initial notch-to-depth ratios

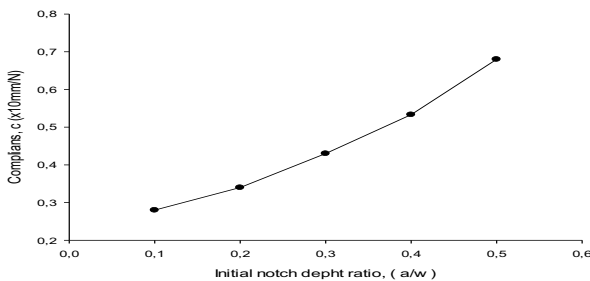


Figure 8. Compliance versus initial notch-to-depth ratio graph of the HNSP 0-50 10% reinforcement

In Figure 9, the results of three different fracture methods of the composites are compared. Among three methods, the J-integral method yielded lower fracture toughness values in the additive concentrations, in which the material is susceptible to fracture, when compared to the other methods. This suggests that the J-integral method is not suitable for the use in fragile composites. Although the other two methods yielded closer values at all the additive concentrations, it can be stated that the compliance method yielded slightly higher values.

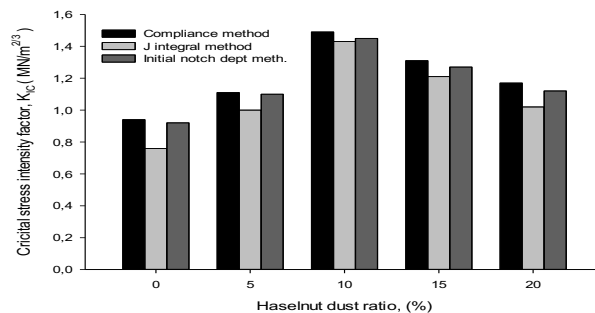


Figure 9. Critical stress intensity factor versus HNSP reinforcement concentration by using J-integral, initial notch depth, and compliance methods

Morphology

SEM micro-images of the pure PMMA (Büyükaya 2017) and the PMMA composites containing 5, 10, 15, and 20% HNSP with particle size of 0-50µm are presented in Figures 10 a, b, c, d, and e. When the fracture prints on the surface are analyzed carefully, it can be easily seen that the pure PMMA did not show a significant plastic deformation. In the fracture surface image of PMMA composite with 5% HNSP additive, it can be seen that the fracture is a brittle fracture but slightly more plastic than the pure PMMA. In the image of PMMA composite with 10% HNSP additive, it can be seen that the fracture is more plastic, whereas the image of PMMA composite with 15% HSNP showed that the plasticity decreased and the breaking behavior became more brittle. The fracture surface image of PMMA composite with 20% HSNP additive confirms that the breaking behavior became more brittle when compared to the PMMA composite with 15% HNSP. This transition between relatively plastic and brittle breaking behaviors reached at the optimum in the sample containing 10% HSNP and the fracture toughness was found to be higher than the other

samples. The internal structure confirms that the fracture toughness is at higher levels in these samples.

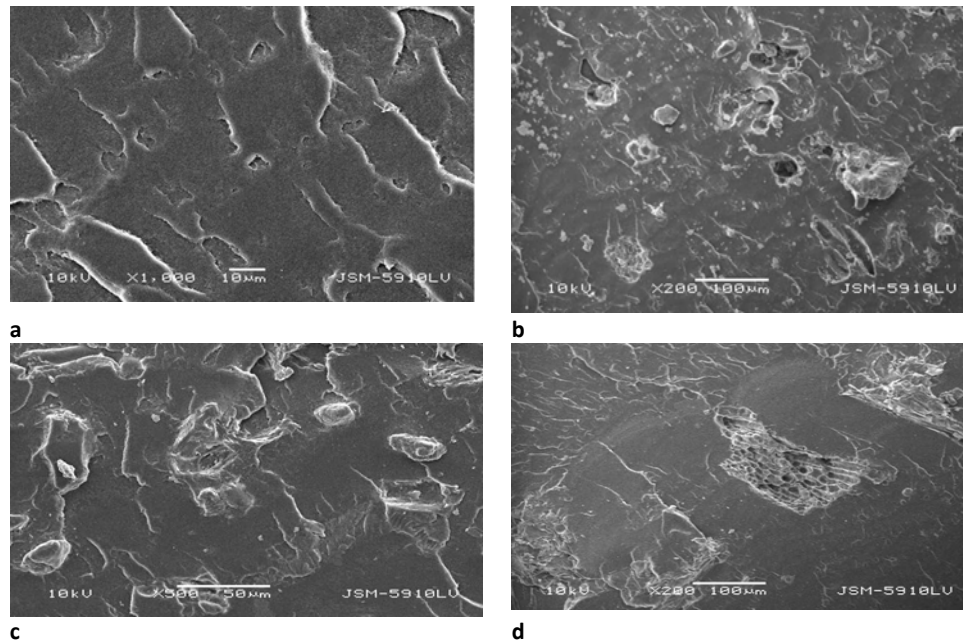


Figure 10 Surface of PMMA (a), 10% HNSD reinforcement (b), 15% HNSD reinforcement (c), 20% HNSD reinforcement

CONCLUSION

The results obtained here showed that the hazelnut shell powder can be used as reinforcement material in composites.

The composites prepared by using this filling material can be used as an environment-friendly product and an alternative to the synthetic materials in structural applications under the effects of fracture.

The composites prepared here were tested using three different breaking methods. According to the results of fracture tests, it was determined that the J-Integral method yielded lower values when the brittle breaking was effective. On the other hand, the initial notch method and the compliance methods yielded very similar results. However, when experimental calibration was applied in compliance method, more reliable results were achieved for the fragile materials.

Whereas the distribution of additive material was more homogeneous in the composites prepared by using hazelnut shell powder at lower concentrations, the agglomeration problem increased at 20% concentration.

These regions constitute weak spots in order for the crack to start.

The production and use of these composites are very important for supporting the awareness of environmentally conscious segments of the society and for more effective use of wastes. Thus, both the social solidarity and economic gains can be achieved.

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