Purnomo
Universitas Muhammadiyah Semarang
Mechanical Engineering Department
Faculty of Engineering
Indonesia

Rudy Soenoko
Professor
Brawijaya University
Mechanical Engineering Department
Faculty of Engineering
Indonesia

Agus Suprapto
Professor
Merdeka University
Mechanical Engineering Department
Faculty of Engineering
Indonesia

Yudy Surya Irawan
Assistant Professor
Brawijaya University
Mechanical Engineering Department
Faculty of Engineering
Indonesia

Impact Fracture Toughness Evaluation by Essential Work of Fracture Method in High Density Polyethylene Filled with Zeolite

The impact fracture toughness of zeolite reinforced high density polyethylene (HDPE) composites which were prepared by an injection molding has been investigated using essential work of fracture (EWF) concept. The Charpy impact tests were carried out on single-edge-notched bending specimens under velocity of 3.4 m s^{-1}. The results showed that the composites with 5 wt.% zeolite had the highest fracture initiation energy because of the toughening effect of zeolite addition. The composites with zeolite content more than 5 wt.% consumed less energy to initiate fracture than that of pure HDPE. However, the energy for propagated fracture decreased by the presence of zeolite and progressively decreased with increasing the zeolite content in the HDPE matrix. This was due to the addition of zeolite particles restricting shear yielding of the composite matrix resulting in lower energy absorption during short impact testing period.

Keywords: *impact, high density polyethylene, zeolite, facture toughness, essential work of fracture.*

1. INTRODUCTION

Cranial implant materials are most commonly used to reconstruct the skull damage due to trauma and tumors. Existing biomaterial implants for skull base recon–struction mostly use alloplastic materials, including polyethylene, hydroxyapatite (HA), titanium mesh, and polymethyl methacrylate (PMMA) [1-5]. Each of them has many disadvantages. Polyethylene and PMMA have low strength and low modulus, the use of PMMA can cause local tissue damage as a result of the heat release during the exothermic reaction [1,2]. Hydroxyapatite is brittle and titanium is heavy, high cost, has poor malle–ability and high heat conductivity. To overcome their drawbacks, Purnomo *et al.* [6] have investigated the fracture behavior of natural zeolite-filled high density polyethylene (HDPE) as an implant skull recon–struction. The fracture behaviour of zeolite-filled HDPE was investigated in quasi-static state using the essential work of the fracture (EWF) method. Natural zeolite was chosen as the filler based on the consideration that it has all the requirements as skull reconstruction implants, i.e., non-toxic and bio–compatible [7], bioactive [8-10], able to protect the HDPE matrix against ultraviolet degradation [11] and an abundant natural resource with low prices. For load-bearing applications such as skull implants that protect the brain, it is important to understand its impact fracture behavior because head injury and skull fracture caused by impact force was the most frequent cause of death.

Recently, the EWF method that originally proposed by Broberg [12] has been progressively used to evaluate the fracture toughness of polymer matrix composites [13-15]. The total fracture work of a specimen having a sharp crack (W_f) is partitioned into EWF (W_e), *i.e.,* the work required to create new surface in its process zone and the non-EWF or plastic work (Wp),*i.e.*, associated with the work done by various deformation mechanisms in the plastic zone and volume-related. Thus, the W_f can be expressed as:

$$W_f = W_e + W_p . \quad (1)$$

$$W_f = w_e.t.l + \beta w_p.t.l^2 . \quad (2)$$

$$w_f = w_e + \beta w_p l . \quad (3)$$

where w_f is the specific total work of fracture, w_e and w_p are the specific essential fracture work and specific plastic work, respectively, l is the ligament length, t is the specimen thickness, and β is a geometrical shape factor of the plastic zone. In accordance with the EWF method, the W_f value is the area under the load-displacement curve. The w_e determined by extrapolation of w_f as a function of l to zero ligament length. The non-essential fracture work (βw_p) were the slope of the linear regression line.

Polymer composites used in skull implants applications are often subject to impact loading. Thus, it is very important to understand the knowledge of their fracture behavior under impact loading conditions. For ductile polymer composites, the EWF methodology can be used for better characterization of the impact fracture

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Correspondence to: Purnomo
Universitas Muhammadiyah Semarang,
Jl. Kedungmundu Raya No. 18, Semarang-Indonesia
E-mail: purnomo@unimus.ac.id
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energy [16-19]. After Wu *et al.* [17] extended the EWF concept for impact testing of ductile polymer blends and Martinatti and Ricco [19] reported that EWF method was valid to assess high rate fracture toughness of polypropylene-based material, many researchers [15-16, 20-23] used the EWF method to characterize impact properties of ductile polymer and polymer composites. This study aims to investigate the impact fracture toughness of zeolite-filled high-density polyethylene using the EWF concept. Furthermore, fracture initiation and propagation energy were determined and discussed based on the results of Charpy impact test and the fracture surface morphology which was observed using scanning electron microscope (SEM).

2. EXPERIMENT METHOD

2.1 Materials and samples preparation

The pellet HDPE used as a thermoplastic matrix polymer was supplied by PT. Lotte Chemical Titan Nusantara Indonesia. The HDPE in pellet form is converted into powder form through a mechanical process and then sieved to 80 mesh size. The natural zeolites obtained from Malang, East Java, Indonesia was used as a reinforcement. In a wide range of particle sizes, they were calcined at 300°C for 3 hours and then cooled by opening the oven so that the zeolite in direct contact with atmospheric air. The zeolite and HDPE powder were mixed in a dry state with the zeolite weight percentage of 5 wt.%, 10 wt.%, 15 wt.%, and 20 wt.%.

Single edge notched bending (SENB) specimens with dimension of 80 mm x 17.5 mm x 3 mm (Figure 1) were formed by injection molding techniques. Injection process was performed at barrel temperature of 160°C with holding in barrel for about 2 minutes. The melt-flow direction was parallel to the longitudinal direction of the specimens.

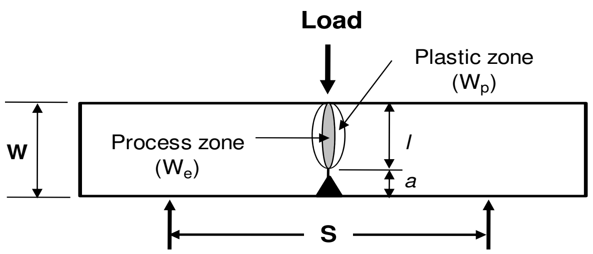

Figure 1. SENB specimen geometry for the EWF method

2.2 Impact essential work of fracture

The notches were made first on the injection-moulded samples by the formation of saw cut, and then they were sharpened with a fresh razor blade. The **ligament lengths were varied of 9, 10.5, 12, 13.5 and 15 mm**. The notched Charpy impact test were performed according to ASTM D 256. The test was conducted using **an impact tester (Gotech GT-7045-MDH)** at impact velocity of 3.4 m s^{-1} at room temperature. The total impact specific work of fracture (w_f) were obtained from the test data presented on the data display on machine. Four samples for each variation of ligament length were tested and average values were reported.

2.3 Scanning electron microscopy (SEM)

The fracture surface morphologies of the SENB specimens were examined by scanning electron microscopy (FEI Inspect S50). All samples were coated with thin layer of Au-Pd prior to SEM observations.

3. RESULTS AND DISCUSSION

During the test, all the SENB specimens were broken into two halves. The fracture energy is the sum of each type of energy that can be absorbed during the Charpy impact test. Figure 2 show plots of the specific total work fracture (w_f) against the ligament length for all specimens tested. For all specimen tested, it is apparent that the specific fracture energy increased with increasing ligament length. In other words, the specific fracture energy depends on the ligament length. All the linear regression plots are showing positive slope associated with non-essential energy term. This indicates that in each composite, not all the fracture energy dissipated in the inner process zone, but there was energy that is dissipated in the outer fracture process zone during impact loading.

According to EWF methodology, the intercept at zero ligamen length and the slope of plot of Figure 2 are the essential and non-essential work of fracture, respectively. The effects of zeolite content on the non-essential work of fracture for all specimens tested are plotted in Figure 3. It is clearly seen that the non-essential fracture work reduced with increasing zeolite content therefore crack propagates faster with little energy absorption. The non-essential fracture work is associated with the energy dissipated by various deformation mechanisms in the plastic zone. During the examination of impact fracture surfaces by SEM, plastic deformation does not take place outside the fracture process zone therefore no stress-whitened zone can be observed in all the fracture samples tested (Figure 4), although all the regression line in Figure 2 shows positive slope. This implies that the energy was dissipated by plastic deformation of matrix about the fracture plane [24].

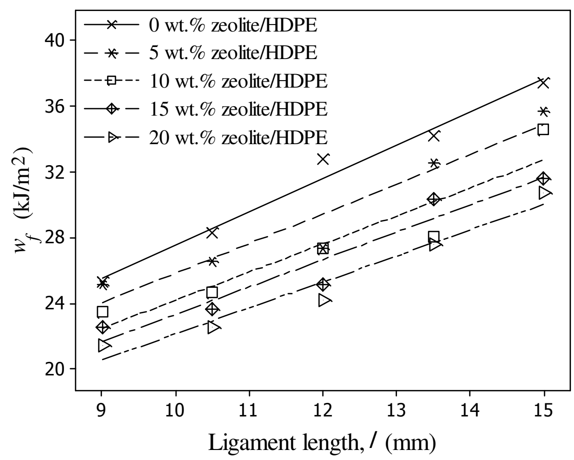

Figure 2. Specific total work of fracture plotted against ligament length for all specimens tested

The essential work of fracture is presented in Fig. 3. It is apparent that the essential work of fracture was increased by presence of 5 wt.% zeolite in HDPE matrix and then it decrease by the addition of zeolite further. However, with the addition of more than 5 wt.% zeolite, the essential fracture work is reduced by increasing of the zeolite content. According to the essential work of the fracture concept, the specific EWF (w_e) and specific non-EWF (βw_p) mean the energy consumed to initiate and propagate the fracture, respectively. Therefore, the fracture initiation energy of 5 wt.% zeolite/HDPE composite is higher than that of both pure HDPE and other composites which their fracture initiation energy were lower than that of pure HDPE and progressively decreased with increasing zeolite content. On the other hand, it is apparent that fracture propagation energy de–creases with increasing zeolite content in HDPE matrix. This means that it was relatively hard to initiate fracture on the 5 wt.% zeolite/HDPE composite but once fracture initiated, the pure HDPE was more resistant than 5 wt.% zeolite/HDPE composite against fracture propagation. These fracture toughness characteristics are similar to the results of the research conducted by Zhang et al [25,26]. They reported that the impact strength increased with filler content increased up to about 6 wt.% but decreased with filler content increased further.

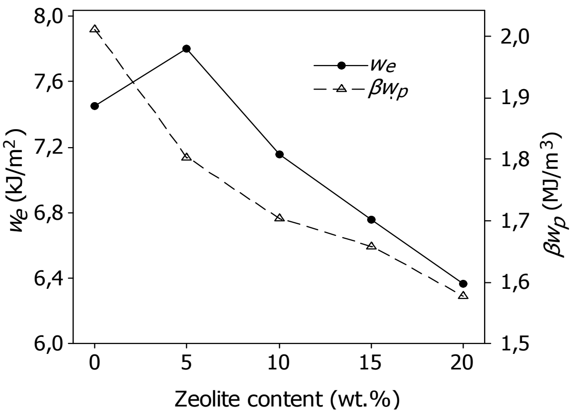

Figure 3. The w_e and w_f as function of zeolite content

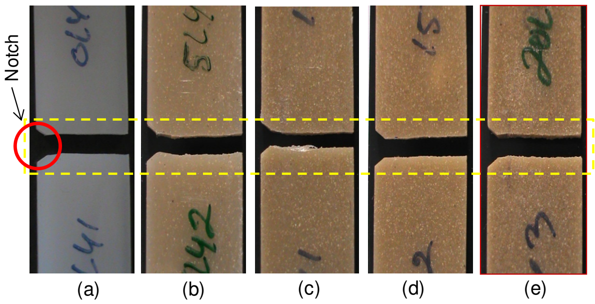

Figure 4. The SENB broken samples for (a) HDPE, (b) 5 wt.% zeolit/HDPE, (c) 10 wt.% zeolit/HDPE, (d) 15 wt.% zeolit/HDPE, and (e) 20 wt.% zeolit/HDPE

The increasing value of we at zeolite content of 5 wt.% probably caused by the diffusion of HDPE into the zeolite particle thereby increasing the interfacial bonding between HDPE and zeolite. The toughening effect is not controlled by the particle fraction [27]. In other words, the fracture toughness is not dependent on the content of the particles. Bartczak et al [28,29] reported that the toughness of the composites was highly dependent on the plastic extensibility of the matrix material. Based on the results of their study, it can be concluded that the distance of interparticle is a property of the matrix material whose existence can alter the microstructural morphology.

Effectively toughening occurs in the zeolite content of 5 wt.%, hence the HDPE can be made super-tough by addition of 5 wt% zeolite to HDPE. The decrease in the we of composites was usually caused by poor dispersion of zeolite particles which tended to form agglomerates with increasing zeolite content in the HDPE matrix. The interparticle contact in the agglomerates is more sensitive to crack than the particle-matrix interface, and thereby the crack easily propagates because practically no crack initiation energy was needed to break the interparticles bonding [30-34].

The impact fracture surface morphologies of the 5 wt.% zeolite/HDPE and 10 wt.% zeolit/HDPE can be seen in Figure 5 and Figure 6, respectively, where the white arrows indicate the direction of fracture propagation. The SEM micrographs as shown in Figure 5(a) and 6(a) were taken near the notch where fracture initiated. They represent a rough fracture surface compared with the fracture surface shown in Figure 5(b) and 6(b), where SEM micrographs were taken away from the notch tip which was the zone of fracture propagation end. The surface morphologies of those composites near the notch evidently showed numerous line cavitations (seen as dark areas) of HDPE matrices separated by shear yielding of that material. While, the smooth fracture surface depicted in Figure 5 (b) and 6 (b) indicated that very little plastic deformation takes place under high-impact speed loading.

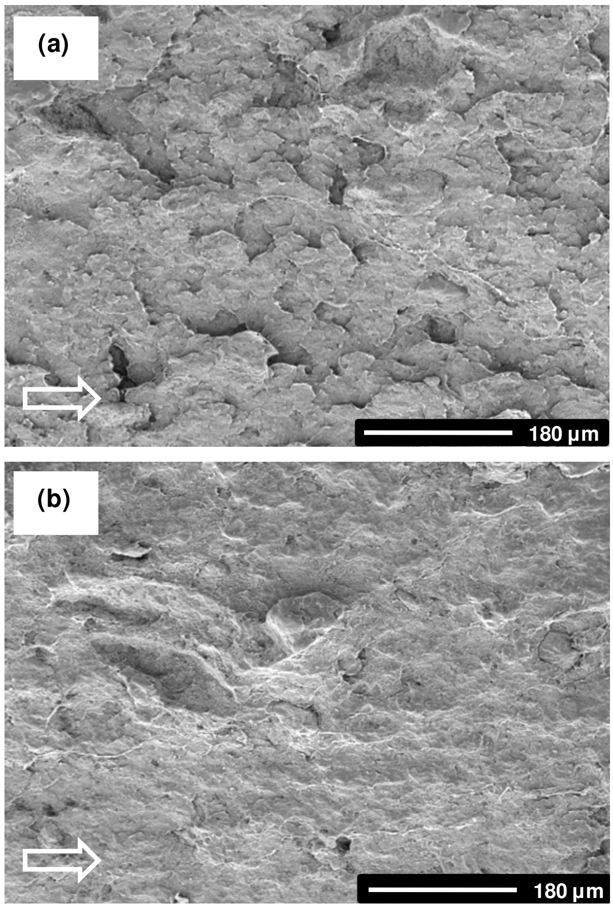

Figure 5. The surface morphology near (a) and away (b) from notch of 5 wt.% zeolit/HDPE

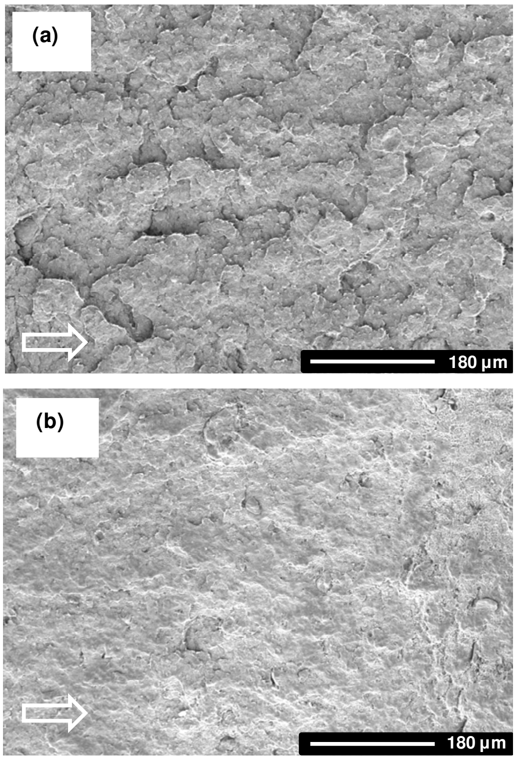

Figure 6. The surface morphology near (a) and away (b) from notch of 10 wt.% zeolit/HDPE

Figure 7 shows the SEM micrographs of 15 wt.% zeolit/HDPE and 20 wt.% zeolit/HDPE near and away from the notch, where the crack propagation direction is indicated by the white arrows. It can be observed that cavitation and debonding occurred on the interface between the zeolite particle and HDPE matrix and their amount reduced in away from the notch tip as shown in Figure 7(b,d). As zeolite content increased to 15 wt.% zeolite (Figure 7), the fracture surface indicating that incorporation of 15 wt.% zeolite to HDPE constraints the yielding of HDPE matrix. A similar fracture feature is observed for the composite containing 20 wt.% zeolite. Hence, higher zeolite content in composite constraints the HDPE matrix from yielding during impact loading under velocity of 3.4 m s^{-1}.

The SEM micrographs of the fracture surface taken on the middle of ligament length with greater magnification was shown in Figure 8 where white arrows indicate the direction of crack propagation in composites. They exhibit variations with respect to zeolite content. The morphologies with line cavitation were present in the fracture surface shown in Figure 8 (a, b). On the other hand, the SEM micrographs also showed the zeolite particles pull out from HDPE matrix resulting from weak bonding at particles-matrix interface and debonding resulted cavitation (Figure 8c, d). All fracture surfaces showed that all composites deformed by forming fibrils occurring during impact fracture. The morphology with fibrillar was probably formed by shear yielding HDPE matrix, which was the mechanism of energy absorbing [35, 23].

In the case of the SEM micrographs as shown in Figure 5(a), 6(b) and 8(a, b), the line cavitations which look as deep furrows were evidence of extensive shear failure connecting the adjacent crack fronts of different planes in HDPE matrix composites [36,37]. An increased-number of cavitations and debonding presented in surface morphology are shown in Figure 5(b), 6(b) and 8(c, d), it can be elucidated that the cavitation mechanism of rigid particles consists of stress concentration at the crack tip, debonding at the particle-matrix interface and shear yielding of the matrix [26, 38]. Debonding at these mechanisms is needed to initiate matrix shear yielding by altering the stress state of host matrix surrounding the voids [26].

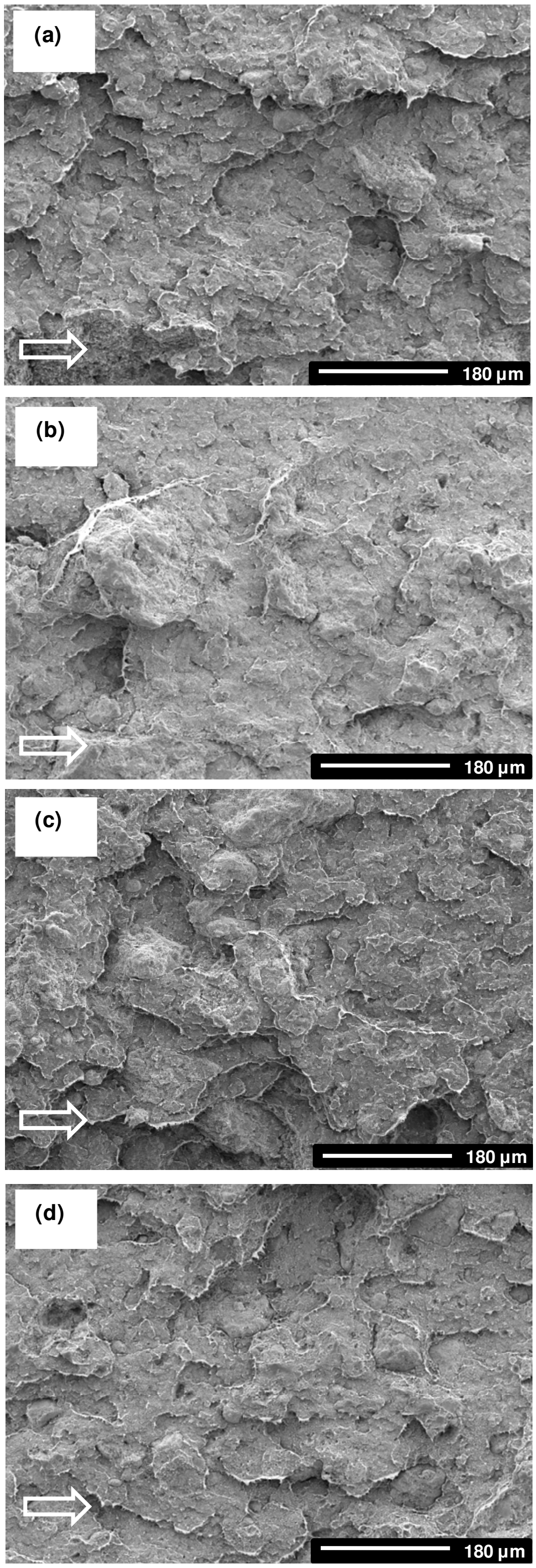

Figure 7. The SEM micrograph of 15 wt.% zeolite/HDPE (a,b) and 20 wt.% zeolite/HDPE (c,d), wherein (a) and (c) are near the notch, while (b) and (d) are away from notch

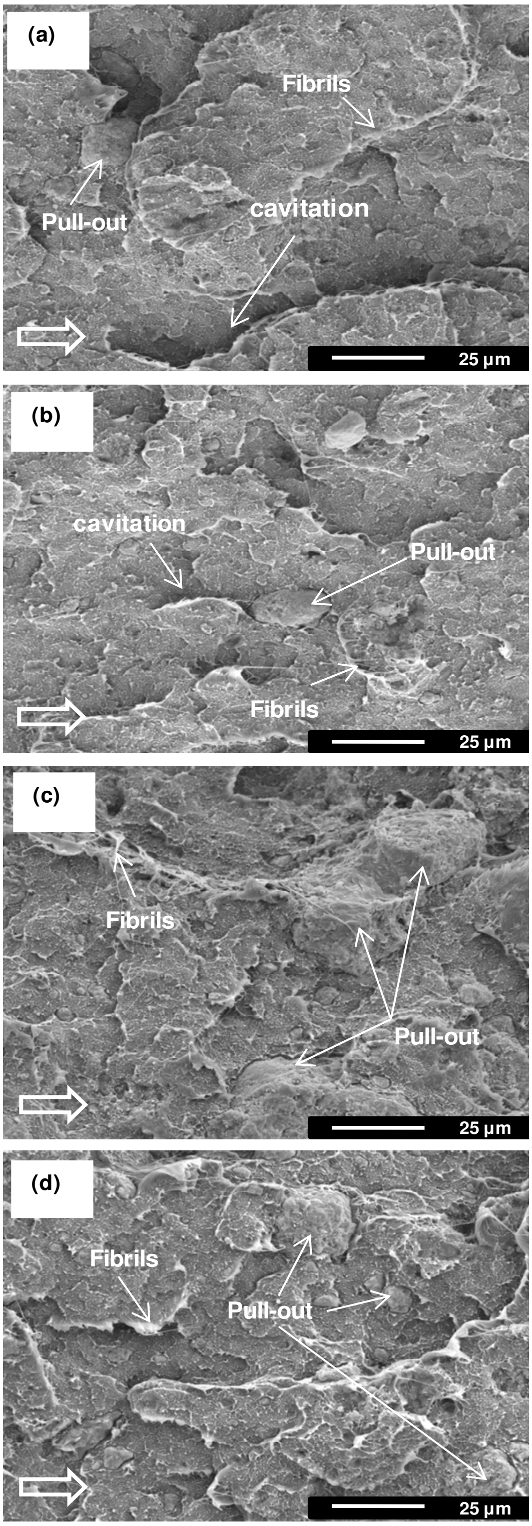

Figure 8. The SEM micrograph of the fracture surface taken on middle of ligament length; (a) 15 wt.% zeolite/HDPE, (b) 10 wt.% zeolite/HDPE, (c) 15 wt.% zeolite/HDPE, and (d) 20 wt.% zeolite/HDPE

Impact fracture behavior as represented by the fracture surface morphology was controlled to a greater extent by factors that influence the fracture propagation initiated by the stress concentration at the notched tip. The addition of zeolite particle led to the creation of particle-matrix interface which constitutes the stress concentration point. Crack propagated easily through the particle-particle interface in the agglomeration which tends to form with increasing zeolite content. Hence, the essential fracture work progressively decreased with increasing zeolit content more than 5 wt.% (Figure 3). In this case, the crack at the interface during impact fracture can be illustrated as shown in Figure 9, by assuming that the shape of the zeolite particles were spherical.

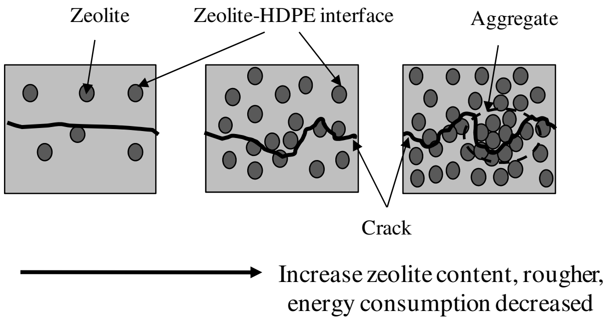

Figure 9. Schematic explanation of crack at the matrix-particle interface during impact fracture in the composites system

4. CONCLUSIONS

The impact fracture toughness of zeolite-filled high density polyethylene prepared with different ratios was investigated by the EWF method using SENB specimens. The Charpy impact test showed that the composite with zeolite content of 5 wt.% represents the material with optimum composition to improve the fracture toughness. However, the presence of zeolite progressively decreased the fracture propagation energy with increasing zeolite content. Examination of fracture surface reveals that the matrix shear yielding, zeolite particle debonding and pull out are responsible for the energy absorbing mechanisms during impact loading.

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ПРОЦЕНА УДАРНЕ ЖИЛАВОСТИ ЛОМА МЕТОДОМ ИНИЦИЈАЛНОГ РАДА ЛОМА КОД ПОЛИЕТИЛЕНА ВИСОКЕ ГУСТИНЕ СА ЗЕОЛИТОМ

Пурномо, Р. Соеноко, А. Супрапто, Ј. С. Ираван

У раду се приказује истраживање ударне жилавости лома код композитног материјала од полиетилена високе густине ојачаног зеолитом, који је припремљен ињекционим обликовањем, применом концепције иницијалног рада лома. Ударно испитивање по Шарпију извршено је тестовима на савијање на узорцима са једним рубним урезом при брзини од 3,4 м/сек$^{-1}$. Резултати су показали да композити са 5 тежинских процената зеолита имају највећу иницијалну енергију лома услед ефекта жилавости који настаје додавањем зеолита. Композитима са садржајем зеолита већим од 5 тежинских процената потребно је мање енергије за настанак лома него чистом полиетилену. Међутим, енергија ширења лома се смањује услед присуства зеолита и прогресивно опада са повећањем садржаја зеолита у матрици полиетилена. Ово је резултат додавања честица зеолита које ограничавају пластичност матрице композита, што доводи до мање апсорпције енергије у кратком периоду испитивања на удар.