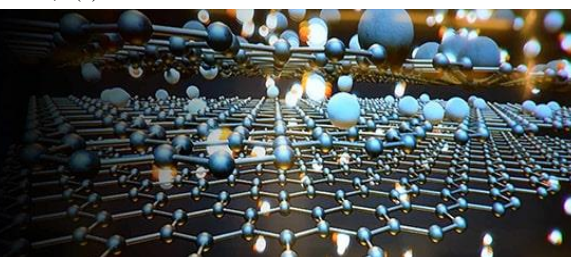


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Methods for increasing plastic deformation of unsaturated polyester by mixing vinyl ester and MMA due to interruption of polymer chain cross-bonds

Nusyirwan Nusyirwan, Abim Pratama and Adam Malik

Abstract

The use of unsaturated polyester as a matrix for making composite materials is very rapidly being used in engineering fields such as automotive components, ship bodies, and aircraft components. One of the weaknesses of this material is that it cracks easily when subjected to impact loads. To improve the crack resistance properties of unsaturated polyester, this was done by adding vinyl ester and MMA diluent and adding MEKP catalyst with the right composition. To evaluate whether the mixed material shows a material that is resistant to cracking, the test refers to the D5405 standard crack test. An important factor that was revealed is that this vinyl ester and polyester blend is a material that is resistant to cracking. For this reason, the results of the fracture of the material after the crack test were observed with an SEM microscope. The test showed the shape of the rough surface fracture on the composition of this 30% polyester and 70% vinyl ester mixture showed a material with a very high level of toughness which was very different from pure polyester that had not been mixed. From the crack test, the stress intensity factor will be obtained as a value that determines the level of crack resistance of a polymer material. From the tests obtained for a mixture of 70% polyester and 30% vinyl ester, the highest stress intensity factor was obtained, namely $2.179 \text{ MPa}\cdot\text{m}^{0.5}$ which increased from pure polyester which only $0.762 \text{ MPa}\cdot\text{m}^{0.5}$, an increase of 286 %.

Keywords: Polyester, vinyl ester, fracture toughness, stress intensity factor

Introduction

With the rapid development of technology, engineering construction materials originating from metal components have been replaced by polymer materials that have a lower specific gravity and are easily shaped ^[1]. Some of the advantages of polymers are that they are cheaper than metal materials, are resistant to moisture, and can be combined or joined with other materials ^[2]. Besides the many advantages possessed by polymers, polymers also have many weaknesses, including not being able to withstand high temperatures, used for a long time can become brittle and crack. Various research is developing to increase the usability of plastics, some are developing towards resistance to high temperatures and some are researching the properties of brittle resistance or research on polymer crack resistance ^[3]. The study that will be discussed in this research is the second scope problem, namely how to increase the brittle resistance and crack resistance of polymers. The cause of the cracking in the polymer between them is because the polymer has long molecular chains and at first, it can be easily deformed well within a certain limit and in the end, the deformation stops and changes the polymer to stiff and brittle ^[4]. Many studies have studied the increase in the ductility of polymers, one of which is by substituting polymer molecules with other material molecules, for example adding catalysts, combining or shortening polymer molecule chains by connecting with other atomic chains and another way is combining them with other polymers to reduce their ductility. Another research is to add atoms of the filler material so that the brittleness and cracking properties are reduced ^[2, 5]. Another effort made is to prevent the brittle nature of polymers by adding elastic materials such as synthetic rubber or natural rubber so that the brittle and easily cracked properties of polymers can be overcome so that polymers are easy to deform ^[6, 7]. Various kinds of research are combining two polymer atom chains that are almost similar and compatible, for example combining polyester with vinyl ester so that both of them can extend the ductility and have easily deformable properties so that they are resistant to impact and vibration ^[8]. Various polymers have their advantages and disadvantages, for example, polyester has good properties for hardness and is not resistant

to impact while vinyl ester has good properties for aging but this material is not resistant to scratches, combining these two materials is a study that is easy to do [3, 5, 9]. The research step to be carried out is to mix polyester with vinyl ester for various percentages and then evaluate its brittle and crack-resistant properties [6].

Various studies to increase the use of polymers include research to improve the fracture toughness properties of polymer materials [10, 11]. The work is done by adding other materials as fillers which can combine to prevent the polymer molecules from locking together so that the brittleness of the polymer can be reduced [11]. The addition of a mixture of 60% polyester and 40% vinyl ester increases the tensile strength to 36 MPa and pure polyester to only 22MPa. The increase in tensile strength was caused by an increase in the composition of the rigid structure of the vinyl ester aromatic benzene ring at high VE compositions [12]. Various polymers have their advantages and disadvantages, for example, polyester has good properties for hardness and is not easily deformed so it is not resistant to impact. Whereas vinyl ester has good properties for toughness but is not resistant to scratches. For this reason, it is necessary to combine these two materials to obtain material properties that are tough and have good hardness [3]. Research stage to be carried out is to mix polyester with vinyl ester with various percentages and then evaluate their brittle and crack-resistant properties [14, 15, 16]. From several previous studies, no information studied the increase in crack resistance of this unsaturated polyester mixed with vinyl ester. Due to a large number of uses of polyester in the engineering field, it is necessary to improve some of its weaknesses, including increasing the toughness and crack resistance of the polymer so that the use of this polymer is very wide [17, 18, 19]. The study that will be discussed in this research is how to increase the cracking resistance of polymers. The steps to be taken are to increase the brittle properties and increase the cracking resistance of polyester (UP) by mixing polyester with the vinyl ester (VE). So to find out about increasing the crack resistance of polyester, it is mixed with vinyl ester with a certain percentage, several tests are carried out including crack testing, FTIR observations, and the shape of the fracture surface will be related to the magnitude of the stress intensity factor value obtained from the crack test. The most important study that will be carried out in this study is to identify the ability of polyester polymers to withstand fracture loads after mixing with vinyl ester [20]. A value that evaluates how much the polymer's ability to withstand cracking loads will be determined by how much the value of the K_{Ic} crack toughness factor is obtained [8, 21, 17].

Experimental Section

Materials

Some of the materials used in this study include polyester, vinyl ester, MMA, and MEKP. Polyester is a polymer that is widely used as a matrix to form polymer composite materials or as a binder or fiber covering in making composites for construction in the form of plates or flat surfaces. Polyester has the advantages of being a polymer that is easily shaped, scratch resistant, and includes a cheap

polymer material [2]. The following are some of the properties of polyester, including polyester has relatively high tensile strength, resistance to changes in weather, chemicals, and mold, excellent abrasion resistance, easy maintenance, and polyester has water-repellent properties, and dries quickly [22, 16, 17]. The weakness of polyester is that it cracks easily and breaks easily which is part of this research. The type of polyester used in this research is unsaturated polyester with Yukalac1560 BL-EX product. The mechanical properties of polyester can be seen in Table 1.

Table 1: Mechanical properties of polyester

Item	Unity	Value
Tensile strength	MPa	20-100
Tensile modulus	GPa	2.1-4.1
Ultimate strain	%	1-6
Poisson's ratio	-	-
Density	g/cm ³	1.0-.45
T _g	°C	100-40
CTE	10 ⁻⁶ /°C	55-100
Cure Shrinkage	%	5-12

The polymer vinyl ester polymer has good elastic deformation compared to polyester but has a relatively expensive price compared to polyester, vinyl ester is produced by PT. Justus Kimiaraya with the trademark vinyl ester resin.

Methyl methacrylate or often referred to as MMA is a polymer material that has biocompatible properties. The advantages of adding MMA to polyester alloys are that it produces materials that are non-toxic, relatively low cost, easy to process, compatible, and can be used to process materials that have good crack resistance [24]. Mixing MMA with polyester can reduce the viscosity of the polymer mixture and can homogenize the polymer [3]. Catalyst used is Mepoxe catalyst produced by PT. Justus Kimiaraya. The function of the catalyst is as a catalyst to accelerate the drying rate of polyester. The use of catalyst is 4% for polyester alloys.

Prepare composition of a mixture of polyester and vinyl ester

In this research, the manufacture of polymer material mixed with polyester and vinyl ester polymer with the following ratio of polyester to vinyl ester 100%: 0%, 70%: 30%, 30%: 70%, and 0%: 100%, each mixture will be compared with the mechanical properties of all the percentages of the mixture made and will be compared with pure polyester without being mixed with vinyl ester. This section describes the steps for making polymer blends between polyester and vinyl ester Standar ASTM D5405. The magnitude of the stress distribution that occurs at the crack tip for the material given the initial defect is called the stress intensity factor. The magnitude of the stress intensity factor of a material with certain dimensions against a given load until the material experiences a complete crack is called the critical stress intensity factor, this term is known as fracture toughness which is denoted by (K_{Ic}).

Table 2: Composition of the Crack Test Sample of a mixture of polyester and vinyl ester.

Material No.	UP Composition (wt %)	Vinyl Ester Composition (wt %)	MMA Composition (wt %)	MEKP Composition (wt %)
1	100	0	10	4
2	70	30	10	4
3	30	70	10	4
4	0	100	10	4

Homogezening blends of polyester and vinyl ester

The hot plate hot plate magnetic stirrer is used as a stirrer for the mixed matrix and reinforcement with a temperature that can be adjusted as desired so that the composite material is well formed. The specifications of the hot plate magnetic stirrer are as follows; Brand Daihan Scientific, Model MS-H280-Pro, Working Temperature 25-280 °C, rotation 0-1500 rpm. The process of making mixed

materials is done by pouring a homogeneous or uniform mixture into the mold and then pressing it evenly with a roller or brush, this process is repeated until the required thickness is reached. The test specimen will be made according to the dimensions which refer to the standard dimensions following the ASTM D5405 standard as shown in Fig. 3 [25, 26].

Specimen freezing mould

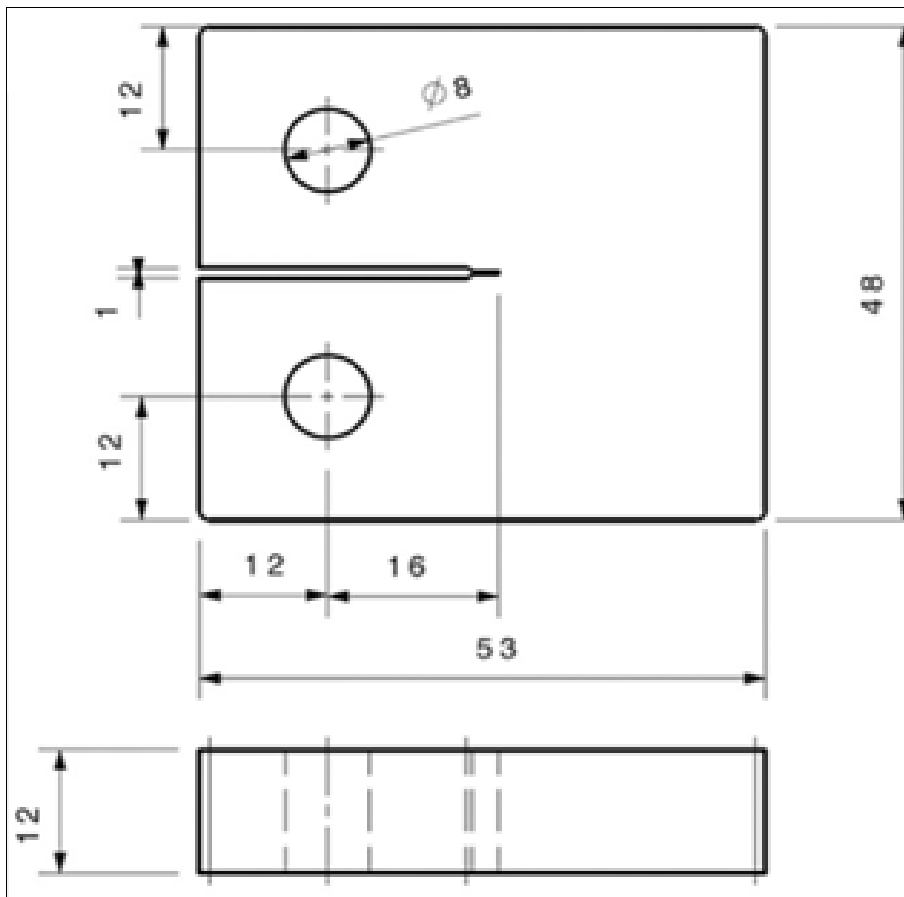


Fig 1: Dimensions of Crack test Sample According to ASTM D5405 [25].

The process of making mixed materials is done by pouring a homogeneous or uniform mixture into the mold and then pressing it evenly with a roller or brush, this process is repeated until the required thickness is reached. The test

specimen will be made according to the dimensions which refer to the standard dimensions following the ASTM D5405 standard as shown in Fig. 1.



Fig 2: Example of casting result from crack test sample according to ASTM D5405.

Crack testing machine

The test carried out is a crack test using a crack testing machine referring to the ASTM D5405 test standard. The crack testing machine is used for crack testing of composite

samples that have been made, according to the referenced standard, namely ASTM D5405, the dimensions can be seen in Fig. 1. This tool will be able to input material specification data needed for the purposes of analysing the

crack strength properties of composite material samples. The specifications of the crack testing machine are as follows; Brand COM-TEN testing machine 95T Series 5K,

Capacity 5000 Pounds, load Cell Model TSB0050, with a touch screen monitor display system or com-touch screen.

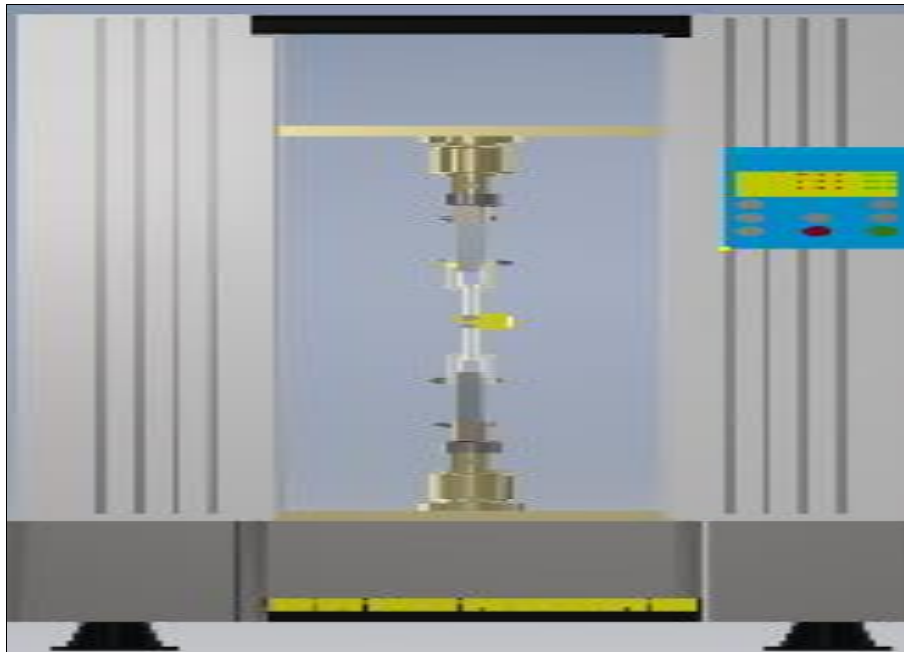


Fig 3: Crack testing machine Brand merk COM-TEN according to ASTM D5405.

The testing was carried out using the two-sided vertical tensile loading method with an adjustable withdrawal speed to provide a good pulling effect of a maximum of 4 mm/minute, if the speed exceeds 4 mm/minute it will pose a risk of dynamic effects. In this study, the mixed materials were made according to what is written in Table 2. Each mixture will be evaluated for the force generated by the machine to provide a tensile force until the material breaks completely and compared with the crack strength of all the percentages of the mixture made and the last will be compared with pure polyester without mixture. This section describes the steps for preparing specimens of polyester and vinyl ester blends. The crack testing machine is used for crack testing of composite samples that have been made, according to the referenced standard, namely ASTM D5405, the Crack testing Machine can be seen in Figure 3.

$$K_{Ic} = \frac{P}{BW^{1/2}} \cdot f\left(\frac{a}{w}\right) \quad (1)$$

$$f\left(\frac{a}{w}\right) = \frac{\left(2 + \frac{a}{w}\right) \left\{ 0.886 + 4.64\left(\frac{a}{w}\right) - 13.32\left(\frac{a}{w}\right)^2 + 14.72\left(\frac{a}{w}\right)^3 - 5.6\left(\frac{a}{w}\right)^4 \right\}}{\left(1 - \frac{a}{w}\right)^{3/2}} \quad (2)$$

Interpreted as

K_{Ic} = Stress intensity factor (MPa.m^{0.5})

P = Maximum load (kN)

B = Specimen thickness (cm)

W = Specimen width (cm)

a = Crack length (cm)

Results and Discussion

Fourier-transform infrared (FTIR) Spectroscopy Analysis

The mixed polyester and vinyl ester materials were evaluated using Fourier-transform infrared (FTIR) spectroscopy analysis to determine the OH hydroxide functional groups formed from the polymer mixture for each mixture. In this study, the polymer mixture was prepared from a mixture of polyester and vinyl ester. FTIR is used to qualitatively analyze the characteristics of functional groups in a chemical compound present in pure polyester samples and a mixture of Polyester and Vinylester. Functional group scanning with materials with variations in the percentage of mix observed was 100% pure polyester, and vinylester mixtures, namely 70% polyester/30% Vinylester, 30% Polyester/70% Vinylester, and 100% Vinylester. The UP/VE mixture was tested with a SHIMADZU brand FTIR tester. The figure shows the FTIR spectra of the UP/VE mixture at various cured VE compositions at room temperature. This is observed from Figure 4. FTIR analysis, the peak readings at 1471.69 and 719.45 cm⁻¹ correspond to the C-H para-substituted aromatic vinyl ester benzene ring. The peak at 2914.44 cm⁻¹ increases with the formation of long-chain molecular structures. The peak at 939.33 cm⁻¹ decreased according to the reaction of the vinyl functional groups. Figure 4 shows the UP/VE mixture at high VE composition. The peak at 551.64 cm⁻¹ corresponds to the C-H monosubstituted aromatic benzene ring showing high intensity. It shows the rest of the benzene ring and the unreacted molecule.

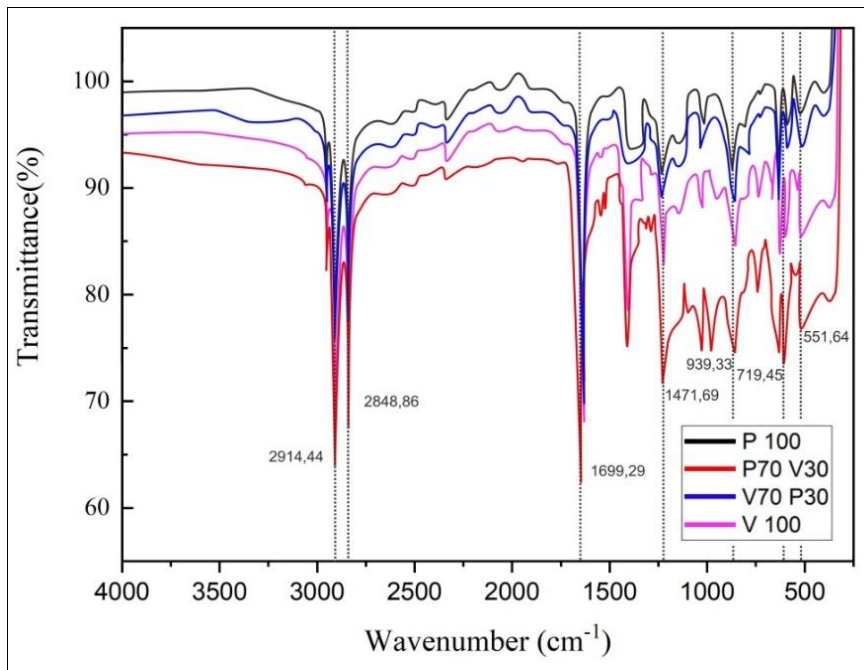


Fig 4: FTIR Curve of Polymer Blends of Polyester and Vinyl Ester.

XRD Curve of Polymer Blends of Polyester and Vinyl Ester

From XRD, identification and phase analysis of a material in the form of polycrystalline and amorphous were obtained. If monochromatic X-rays hit the crystalline material, then each plane of the crystal will reflect or scatter X-rays in all directions. Interference occurs only between reflected rays in phase so that only certain reflected X-rays are present. The interference is mutually reinforcing when the X-rays that are in phase have different paths that are multiples of the wavelength (λ). This statement is called Bragg's law for crystal diffraction. From the analysis of the graphic pattern in Figure 5, the graphic pattern of the polymer mixed polymer specimens of 30% - vinyl ester 70% shows no diffraction peaks, this proves that the specimen has an unstructured diffusion pattern or has random atoms (amorphous), the arrangement of the particles is not dense,

and has the fastest hardening, as shown in Figure 5., so that the material is tougher and more ductile. In other variations of the specimen, it still has an unstructured diffusion pattern or has random (amorphous) atoms, but in the 100% polyester specimen, it appears that there is a slight peak so that it has the firmest properties compared to other materials and is followed by 70% polyester and 30% Vinyl ester, and Polyester 30% and 70 vinyl ester, and vinyl ester 100%. The pattern contained in the XRD curve with a sample variation of 100% vinylester has the highest intensity and 100% Polyester follows below. In the variation of the polyester mixture with the addition of vinyl ester, the intensity value decreases along with the increase in the percentage of vinyl ester in the mixed specimen. This is evidence that vinylester can disrupt the cross-linked network of atomic structures.

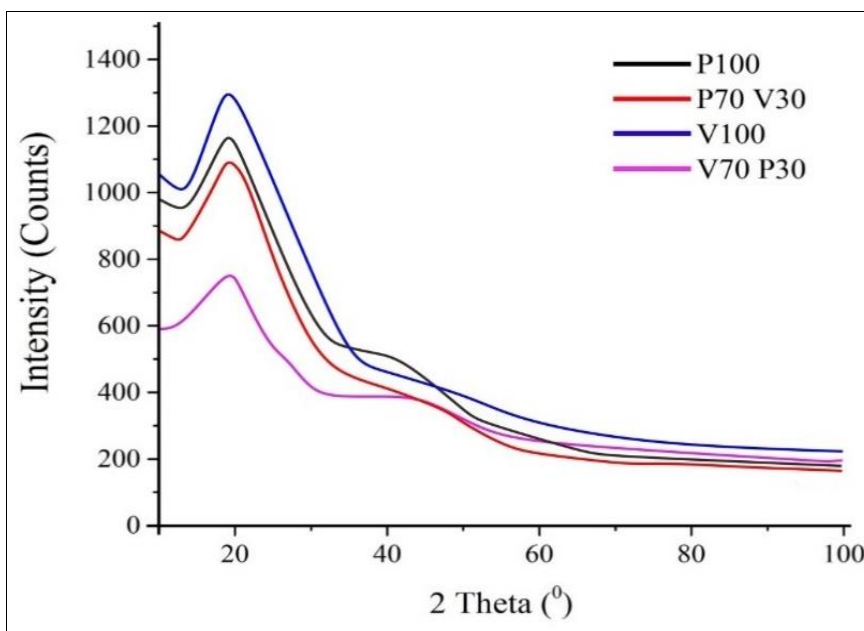


Fig 5: XRD Curve of Polymer Blends of Polyester and Vinyl Ester.

Fracture Surface of Polymer Blends of Polyester and Vinyl Ester

To observe the shape of the surface of the material after experiencing cracks due to the crack testing machine observed with an electron microscope brand FE-SEM from JEOL with a high induction current of 5 kV and a proximity season current of 8 mA. The fracture surface of the material from the test sample was observed with an electron microscope with the FE-SEM brand from JEOL with an accelerating current of 5 kV and a probe current of 8 mA. The fracture surfaces of unsaturated or mono polymer unsaturated polyester fracture samples are shown in Figure 6(a), which has a smooth surface because this material has very little deformation or is a brittle material. Figure 6(b) shows the fracture surface of a pure vinyl ester fracture sample, which has a slightly rougher surface than pure polyester but is still classified as a slightly tough polymer. For the fracture surface, a mixture of vinyl ester on unsaturated polyester produces a rougher fracture surface. Figure 6(c) depicts the characterization of the crack surface for the addition of 30% vinyl ester to polyester (UP/VE 30%), with a rough crack surface forming, indicating that good plastic deformation with the meaning of the word has occurred. In this area, the brittleness of polyester can be reduced, resulting in plastic deformation. the addition of vinyl ester to unsaturated polyester produces a rougher surface and this indicates the material has high toughness

properties. Figure 6(b) depicts the fracture surface characterization for the addition of 70% polyester and 30 % vinyl ester, with three distinct fracture surface forms. This unscratched fracture surface shows the fracture surface area with slow fracture growth. The fracture surface characterization is shown in Figure 6(c) at a 30% polyester and 30 vinyl ester rate. area (1); crack surface area with a smooth cracked surface with only a small number of cracked surfaces. Region (2) is the transitional crack surface area where the rough cracked surface begins to form a region (3). The very rough crack surface has a rougher appearance due to the rapid growth of the radial cracks due to the formation of circular nanovoids (red arrows) and produces different roughness lines [3]. The smooth fracture surface indicates that the material cannot resist the energy from the load exerted by the tensile testing machine (1) results from the slow crack growth and tortuousness along the fault groove, with the lowest plastic regions in the crosslinked polymer chain structure being slightly disturbed. Regions (1) and (2) reflect the characteristic appearance of transitional plastic deformation. The rougher the crack surface, the higher the plastic deformation with characteristic coastal markings as shown in Figure (6c), location (3). With the addition of failure energy, the crack growth rate increases until the collapsed end in area (4) it is shown that there is a circular empty area (nano void) which indicates the roughest cracked area.

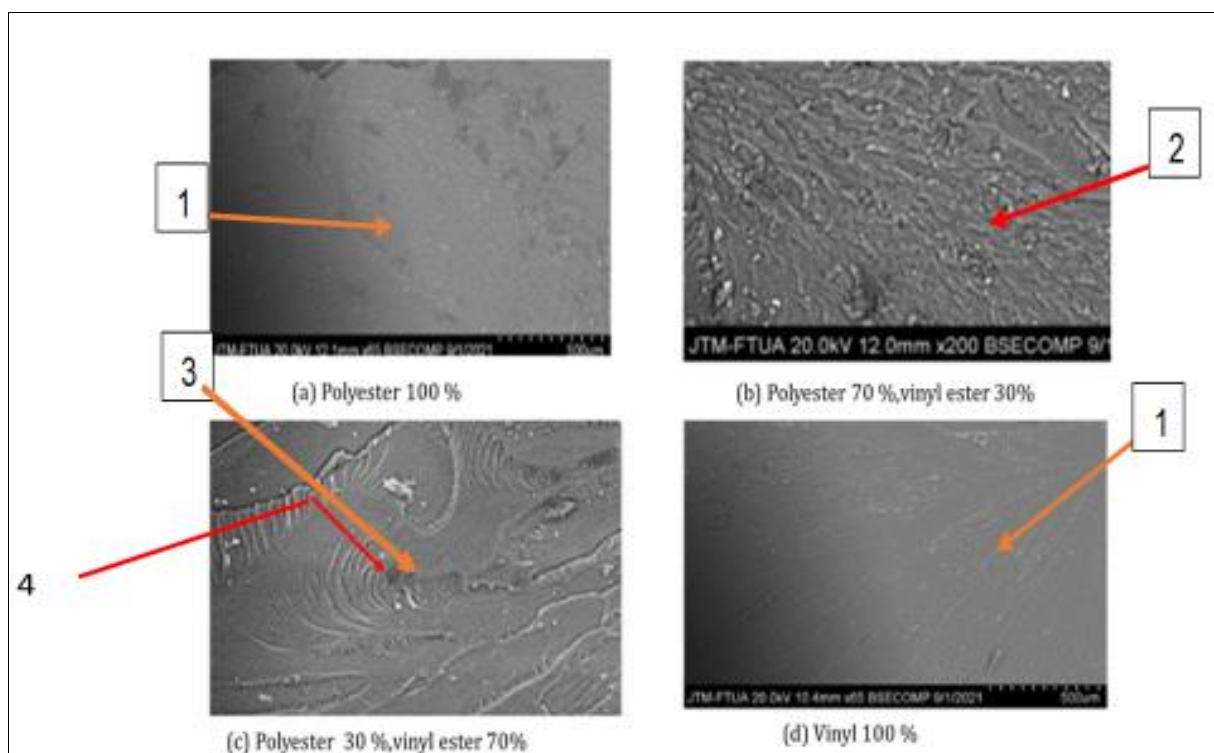


Fig 6: XRD curve of polymer blends of polyester and vinyl ester

Mechanism Toughness Properties of Polymer Blends of Polyester and Vinyl Ester

In the Table 3, it can be shown the magnitude of the force exerted by the crack testing machine to fail different materials for each mixture and Figure 7, shows the magnitude of the force applied by the machine to provide fracture failure samples respectively for pure UU polymer mixtures, pure VE, and UP/VE. From the point of view of crack testing, it is characterized that for brittle specimens,

crack propagation will form a straight line trajectory, while material that is resilient to cracks will form a jagged upward curve not forming a straight line gradient. The load will decrease after reaching the maximum, indicating that the elastic limit of the material has been lost. This shows that most of the energy from the fracture load is absorbed by the material to maintain the fracture force felt in each of the atomic elements that make up the material. After a tensile load is applied, the unsaturated polyester is reinforced with

(30 wt.%). VE undergoes further elongation (displacement) compared to pure UP. This is attributed to cracks initiated at deflected loads due to interfacial adhesion with bonded VE and UP polymers. As the cracking increases, the load continues to increase, as shown in Figure 7. For specimens with up to 70 wt.%, When VE is added to the UP mixture, the VE composition is well dispersed in the UP matrix at the fracture tip location and can reduce the local stress concentration, allowing the mixture to be mixed. It enables the polymer to withstand greater loads and produce more fractures.

In the crack test, it can be observed, the cracked test object shows straight-line propagation for the brittle material in only a few areas in the form of unstable serrations or cracks while the tough material shows a jagged crack pattern. This also implies that most of the energy is absorbed in the initial cracking stage, and a small amount of energy is absorbed in the crack propagation stage. Table 3 and Figure 7 show the magnitude of the force applied by the machine to provide fracture failure samples for the pure UP, pure VE, and UP/VE polymer blends, respectively. Figures 11 show a comparison of the energy consumption for resisting cracking of materials from different specimens. The crack load is presented in Table 3. Representative loads against recorded time curves for samples affected at different energy levels are shown in Figure 8. Load time curves for all laminated polymer is linear until the initial point of

breakdown, then reaches a peak load. After the initiation of damage, the load drops suddenly, indicating a decrease in the stiffness of the material as the polymer matrix fails. The highest load indicates the maximum load that the cracked test object can withstand before experiencing major failure. The highest loads taken by the post-impact samples for the three energy levels, 0.25, 0.63, 0.89, and 0.77 N.mm, showed a sizeable reduction (Fig. 8). The load time curves for all laminated polymers are linear until the initial point of failure, at which point they reach the maximum load. After the initiation of damage, the load drops suddenly, indicating a decrease in the stiffness of the material as the polymer matrix fails. The highest load indicates the maximum load that the cracked test object can withstand before experiencing major failure. The highest loads taken by the post-impact samples for the three energy levels, 0.25, 0.63, 0.89, and 0.77 N.mm, showed a sizeable reduction (Fig. 8). This drastic reduction in the maximum load for the post-crack test specimen is associated with polymer failure as a result of loss of stiffness unsaturated polyester fracture samples are shown in Fig 6(a), which has a smooth surface because this material has very little deformation or is a brittle material. Fig 6(b) shows the fracture surface of a pure vinyl ester fracture sample, which has a slightly rougher surface than pure polyester but is still classified as a slightly tough polymer a probe current of 8 mA.

Table 3: Crack Tensile Load of Test Sample of a mixture of polyester and vinyl ester

Material No.	UP Composition (wt %)	Vinyl Ester Composition (wt %)	Force (N)	K _{1c} (MPa.m ^{0.5})
1	100	0	164.23	0.762
2	70	30	198.46	1.566
3	70	30	499.64	2.179
4	0	100	256.76	1.277

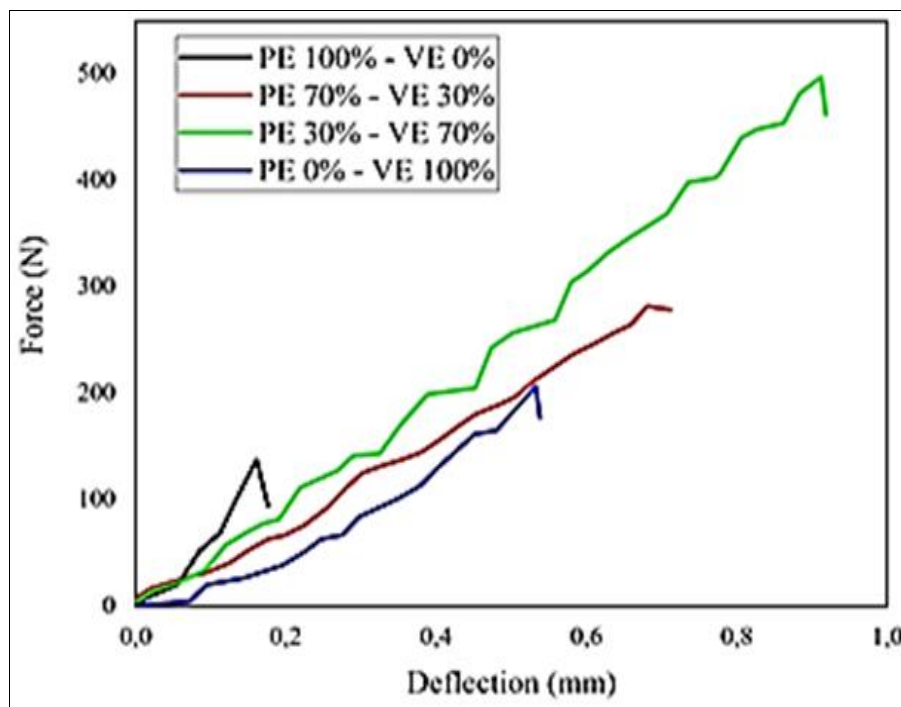


Fig 7: Crack Tensile Load of Polymer Blends of Polyester and Vinyl Ester.

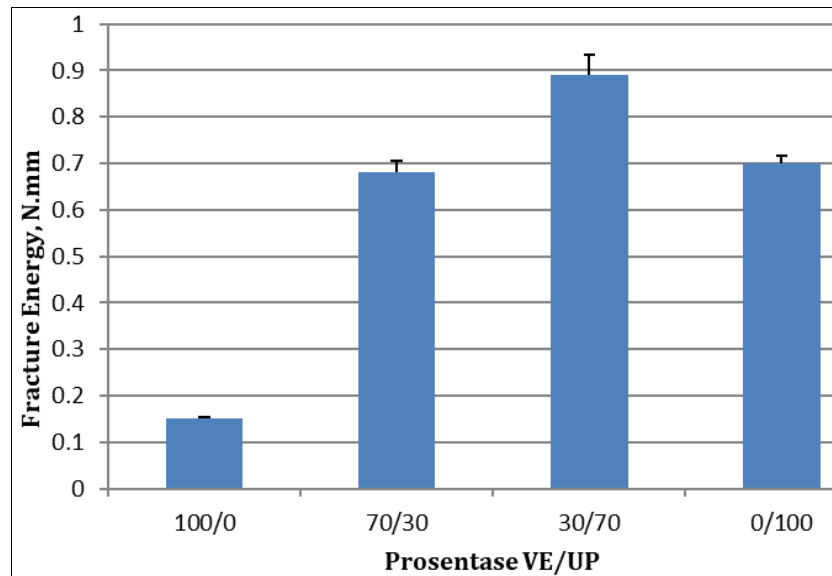


Fig 8: Fracture energy of polymer blends of polyester and vinyl ester

Stress intensity factor of polymer blends of polyester and vinyl ester

From the results of the crack test, it was obtained the maximum crack load that each sample could withstand. The toughness value of the test sample material is obtained by the value (K_{Ic}) for each sample which is calculated by equation (1) and equation (2) the results are shown in Table 3 and Figure 9. In this figure shows the shape of the crack force magnitude curve for the test sample for each percentage of the polyester and vinyl ester mixture given by the crack testing machine. All samples showed different cracking forces depending on the content of the mixture. The test shows that the value of K_{Ic} from each test sample for a mixture of unsaturated polyester mixed with vinyl ester

shows a value that increases with increasing vinyl ester content in the mixture. When adding 70% vinyl ester and 30% polyester, the highest $K_{Ic} = 2.179 \text{ N.m}^{0.5}$, an increase of 286% compared to pure unsaturated polyester $K_{Ic} = 0.762 \text{ N.m}^{0.5}$. The addition of vinyl ester up to 70% increases the value of K_{Ic} due to the emergence of polymers that are resistant to cracking loads which are far superior to the fracture toughness of pure polyesters. The comparison of K_{Ic} values for pure unsaturated polyesters is very different from those for polyesters that have been mixed with vinyl esters. The effect of combining these two polymers can increase the fracture toughness of pure polyester without admixture.

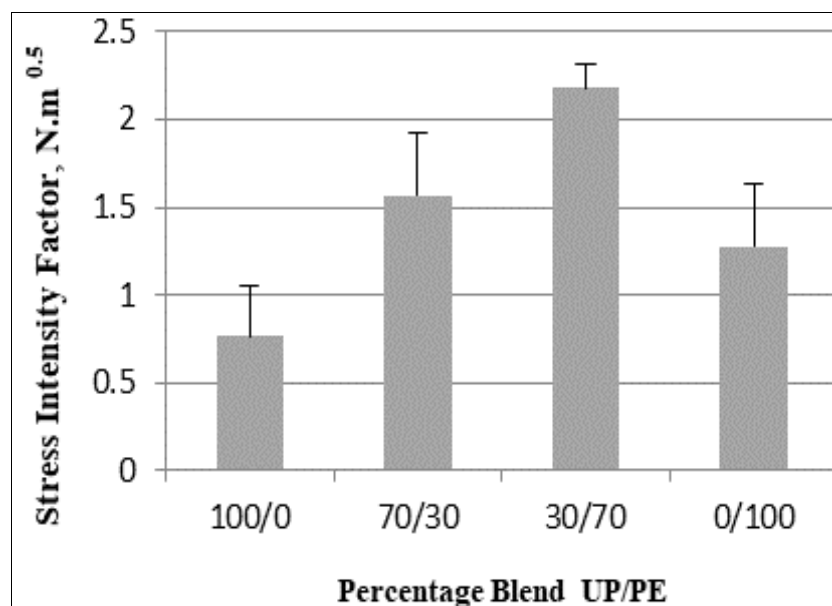


Fig 9: The Stress intensity factor of a mixture of polyester and vinyl ester

Conclusion

This study it was reported the success of changing the brittle nature of unsaturated polyester into a tough material with a large area of elasticity by adding vinyl ester and MMA diluent and adding MEKP catalyst with the right composition. An important factor revealed is that this vinyl

ester and polyester mixed material is a material that is tough against cracking. Tests showed a rough crack surface on a composition of 70% vinyl ester and 30% polyester. The above results can be compared with a mixture of 30% unsaturated polyester with 70% vinyl ester which has the highest critical stress intensity factor resulting in a polymer

mixture that can withstand good cracking loads with a value of $K_{Ic} = 2.179 \text{ N.mm}^{0.5}$ and differs greatly in toughness from pure polyester whose value $K_{Ic} = 0.762 \text{ N.mm}^{0.5}$. The critical stress intensity factor increased from $K_{Ic} = 0.762 \text{ N.mm}^{0.5}$ to $K_{Ic} = 2.179 \text{ N.mm}^{0.5}$ (286% increase) to pure UP. This research can help the engineering field to make a mixture of polyester by adding vinyl ester and catalyst in the right composition, which is a material that is very resistant to cracking.

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Author Contributions

The design of the idea and the experimental design was carried out by Nusyirwan Nusyirwan. Hairul Abral performs control and analysis of test results. Experimental work and data analysis was carried out by Abim Pratama. Crack analysis and material toughness were performed by Hendery Dahlan and writing–review and editing manuscript by Eka Satria

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