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Methods for increasing fracture toughness of thermosetting polyester polymers with vinyl ester mixtures as raw materials for automotive components

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ABSTRACT

Unsaturated polyester is a polymer that is widely used as a basic matrix to form strong composites for engineering applications such as cars, ships, aircraft and other field applications. The advantages of this material are that it has a fairly high tensile strength when reinforced with appropriate reinforcement and is light and easy to shape. However, the weakness of this polymer is that it is brittle and cannot withstand shock loads. To do that, it is necessary to overcome the nature of the weaknesses mentioned above. One of the materials chosen to strengthen polyester with the vinyl ester is because these polymers have almost identical molecular bonds. A crack test was carried out on the addition of vinyl ester with a different composition process. To find out whether this vinyl ester can increase the crack strength of unsaturated polyester and to find out what percentage of the mixture has good crack resistance properties. To determine the value of the crack resistance of the polymer mixture with vinyl ester, a crack resistance test was carried out which would produce a large critical stress intensity factor based on ASTM D 5405 by varying the composition of the polyester and vinyl ester mixture from (0%/100%, 70%/30%, 30%/70% and 0%/100%). From the test results, the greatest critical stress intensity factor occurs in a mixture with a composition of 30% Polyester and 70% vinyl ester of 1.752 MPa.m$^{0.5}$. The critical stress intensity factor increased from 0.762 MPa.m$^{0.5}$ to 2.179 MPa.m$^{0.5}$ (2867% increase) for pure UP.

Keywords: Toughness, polyester, vinyl ester, blends, tensile stress.

1. 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With the rapid development of technology, engineering construction materials originating from metal components have been replaced by polymer materials that have a lower specific gravity and are easily shaped (Abral et al., 2019). 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 (Nusyirwan et al., 2019). 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 researches are 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 (Abral et al., 2021). 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 (Nguyen et al., 2019).

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 (Nusyirwan et al., 2019; Abral et al., 2019). 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 (Hiremath et al., 2020; Liu et al., 2019).

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 (Miao et al., 2020). 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 (Abral et al., 2021; Abral et al., 2019; Rath and Vittal, 2021). 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 (Hiremath et al., 2020).

In this study, the polymer's ability to withstand loads that give a rift effect to the polymer material will be evaluated whether the polymer can crack under a given load at a certain value which is defined as the fracture toughness factor or stress intensity factor or with the symbol Kic (Masoodi et al., 2016). In this study, the value of the Kic stress intensity factor from a mixture of polyester and vinyl ester will be evaluated (Frómeta et al., 2020; Ahmed and Khanna, 2020).

2. RESEARCH METHODS AND MATERIALS

The hypothesis in this study is to determine the polymer mixture of polyester and vinyl ester which has the highest crack strength and determines the level of ability of a strong polymer material to withstand cracking loads which depends on the value of the maximum crack load that the mixed polymer material can withstand.

Material

In this study several types of materials were used including polyester, this material is very widely used in the engineering field because this material is easy to mix and pour into molds because of its easy nature to fill empty spaces so there are no hollow parts. The polyester used is the trademark Yukalac 1560 BL-EX which is produced by the company P.T. Alpha chemistry, with a density = 1.12 g/cm3. The second material is a vinyl ester with the brand name RYPOXY R-802 EX-1, with a density = 1.05 g/cm3. To make it easy for the material to mix between polyester and vinyl ester, methyl methacrylate (MMA) is used and to freeze the material in the mold and also as a catalyst, methyl ethyl ketone (MEKP) is used (Mahyudin et al., 2020; Abral et al., 2019; Albdiry and Yousif, 2019).

Preparation and y Measurement

Unsaturated polyesters (UP), vinyl esters (VE) and methyl methacrylate (MA) are materials used to manufacture tough materials with the compositions (Table 1). The ingredients mentioned in the sub-chapter above after being mixed are then placed in a vibrating pan with the aim that the mixture is homogeneous and completely mixed without any air bubbles trapped in the solution. The tool used is a vibratory pan with the brand Daihan MSH-20D (Natrayan et al., 2022; Mahyudin et al., 2020; Sampath et al., 2021). After uniform mixing then is to print the material at room temperature from 0 to 240 minutes until the material freezes (Frómeta et al., 2020; Abdul-Halim et al., 2022; Klyatskina et al., 2021). The mold of the test material must comply with the material standard for the crack test with ASTM D 5045 standard, with predetermined dimensions, namely 53 mm in length and 48 mm in width and x 12 mm in thickness (Sari et al., 2021; Sampath et al., 2021; Frómeta et al., 2020).

Mechanical Characterization

The casting sample is examined with the fracture toughness test using a universal testing machine COM-TEN (Aynalem and Sirahbizu, 2022; Hunain et al., 2021; Mahmoud-Zaghloul et al., 2021). Testing of specimens based on predetermined standards to provide good crack propagation (Figure 1). The ratio of initial crack to crack width (a/w) is limited to 0.55 and the speed of movement of the load is 5 mm/min. The price for the toughness level of the material to be able to accept the load is determined by the critical stress intensity factor (K1c) calculated according to the test formula from equation (1) and the average value is taken (Arjmandi et al., 2021; Natrayan et al., 2022):

$$K_{1c} = \frac{P_Q}{Bw^{\frac{1}{2}}} . f(\frac{a}{W}) \quad (1)$$

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

The maximum load value can be obtained from the results of the machine crack test curve, the function f(a./w) is a function of equation (2) a polynomial that depends on the shape of the crack propagation of the same material (Miao et al., 2020; Anand et al., 2022; Frómeta et al., 2020).

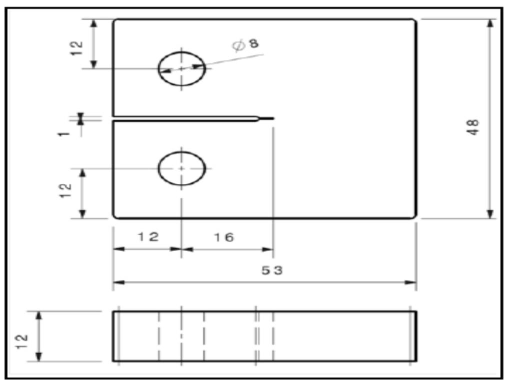
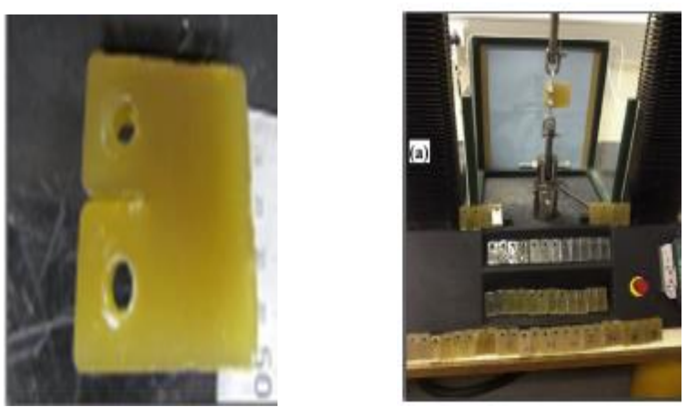


Figure 1 Material dimensions for cracking test according to ASTM D5405 (Klyatskina et al., 2021)



(a) Crack Testing Machine (CTM) (b) Molded Specimens Testing

Figure 2 COM-TEN 95T Series 5K brand crack testing machine

Table 1 Curing Characteristic of UP/VE Blend at Room Temperature

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

3. RESULT AND DISCUSSION

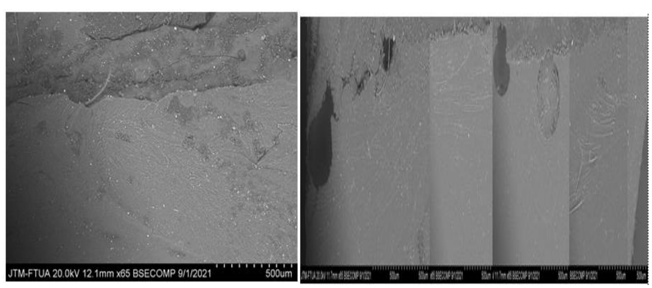
In the following section, we will describe and explain the discussion of research results, analysis of test data and conclusions.

Mechanical Properties of Polymer Blend

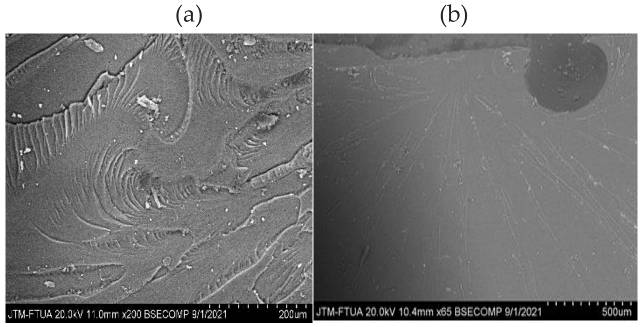
To observe the shape of the surface of the material after experiencing cracks due to the crack testing machine observed with an electron microscope brand FE-SEM from JEOL with a high induction current of 5 kV and a proximity season current of 8 mA (Klyatskina et al., 2021; Gapsari et al., 2022). The 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 (Klyatskina et al., 2021; Gapsari et al., 2022). The fracture surfaces of unsaturated or mono polymer unsaturated polyester fracture samples are in Figure 3(a), which has a smooth surface because this material has very little deformation or is a brittle material. Figure 3(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 (Megahed et al., 2022).

For the fracture surface, a mixture of vinyl ester on unsaturated polyester produces a rougher fracture surface. Figure 3(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 3(d, e) depicts the fracture surface characterization for the addition of 70% vinyl ester to polyester (UP/VE 70%), with three distinct fracture surface forms. This unscratched fracture surface shows the fracture surface area with slow fracture growth. The fracture surface characterization is in Figure 3(d, e) at a 70% UP/VE 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 Nano voids (red arrows) and produces different roughness lines (Abral et al., 2019; Megahed et al., 2022).

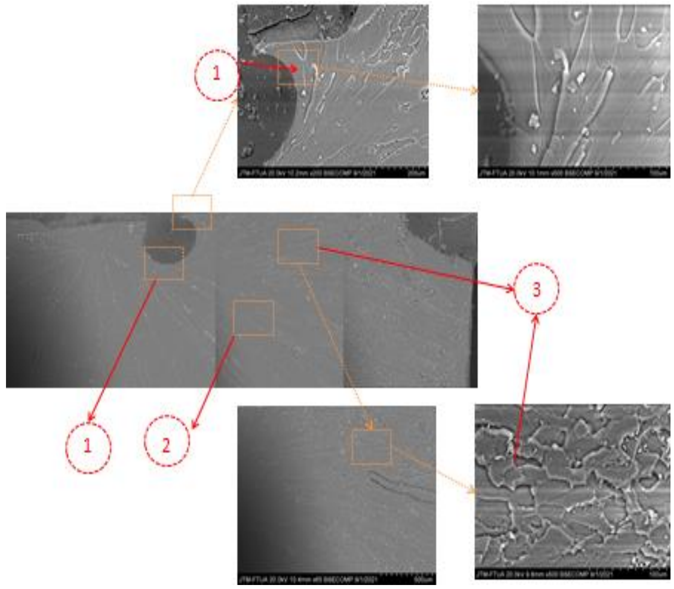
The smooth fracture surface indicates that the material cannot resist the energy from the load exerted by the tensile testing machine (1) results from the slow crack growth and tortuousness along the fault groove, with the lowest plastic regions in the cross-linked polymer chain structure being slightly 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 in Figure 3d, location (3). With the addition of failure energy, the crack growth rate increases until the collapsed end (Moujdin et al., 2022; Frómeta et al., 2020).



(a) (b)



(c) (d)



(e)

Figure 3 The results of the SEM Microscope Test on the fracture surface are shown in (a) UP 100%, (b) VE 100% (c) UP/VE at 70/30%, (d) UP/VE at 30/70 %, (e) Roughest fracture contour

Mechanical Toughness properties

In Table 2 it can be shown the magnitude of the force exerted by the crack testing machine to fail different materials for each mixture and Figure 4 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 (Figure 4). 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 (Bonsu et al., 2022; Klyatskina et al., 2021).

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 2 and Figure 4 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 (Bonsu et al., 2022; Frómeta et al., 2020). Figures 5 and 6 shows comparison of the energy consumption for resisting cracking of materials from different specimens. The crack load is presented (Table 2). Representative loads against recorded time curves for samples affected at different energy levels (Figure 5). 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 (Figure 5). 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 (Figure 5). 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 due to the effects of the post-acting load that caused the crack. Energy absorption is an important factor that is commonly used to assess the ability of polymers to withstand cracking forces. The effect of the crack test response on the absorption magnitude for various percentage crack resistance energy levels (Figure 6). An energy graph showing the amount of energy that must be provided by the crack testing machine to fail the test material and this energy will be absorbed by the test specimen to defend itself so that by providing resistance so that it does not fail and the crack testing machine will provide additional energy until finally the test material is no longer capable give resistance or to the point of breaking. The graph illustrates that the energy resistance level of the material is 27% for the UP/VE mixture, 70/30% and 38% for the UP/VE 30/70% mixture and the lowest is 17% for pure UP and 18% for pure VE. Absorbed energy is compared with all other sample categories (Moujdin et al., 2022; Miah et al., 2022).

Table 2 Load Crack to Fracture Specimens of UP/VE Blend

Percentage UPE - VE (%)	Specimen	Force (kN)	Average (kN)	Standard Deviation
100 – 0	1	0.1370	0.226	0.077
	2	0.2700		
	3	0.2700		
70 – 30	1	0.2820	0.362	0.164
	2	0.2500		
	3	0.5480		
30 – 70	1	0.5210	0.499	0.021
	2	0.4810		
	3	0.4970		
0 – 100	1	0.1780	0.256	0.092
	2	0.2070		
	3	0.3490		

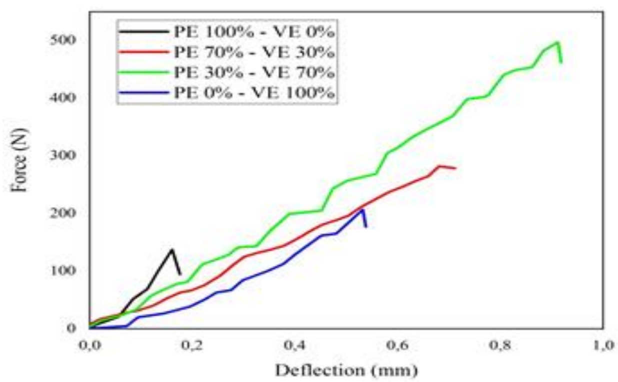


Figure 4 The tensile fracture of UP/VE blends

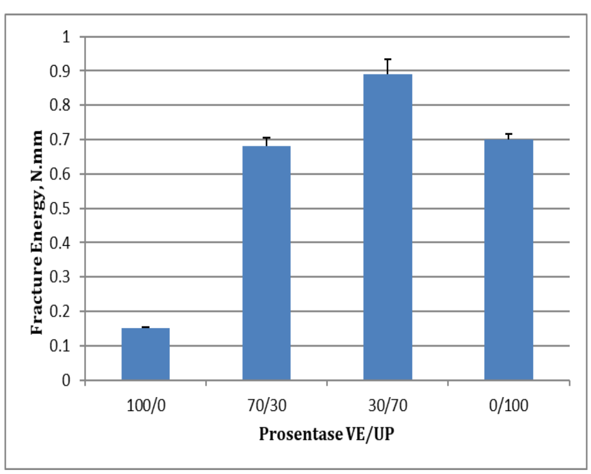


Figure 5 Crack Fracture Energy of UP/VE blends

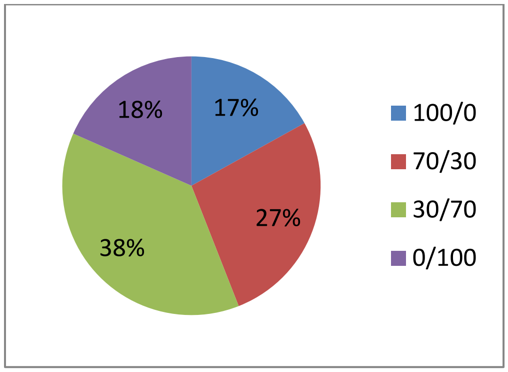


Figure 6 Percentage ratio of Crack Resistance Energy of UP/VE blends

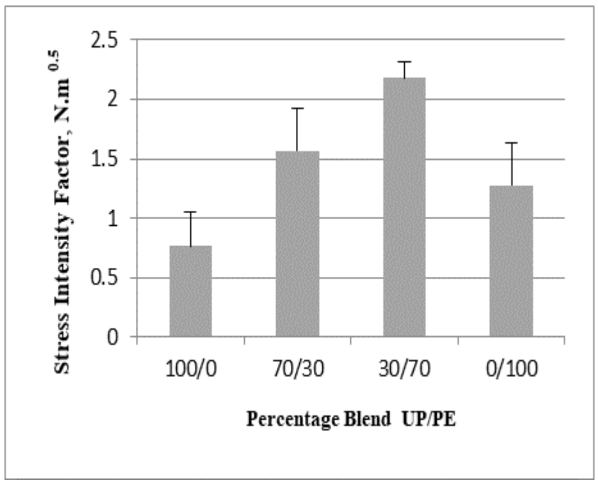


Figure 7 Stress Intensity Factor of UP/VE blends

Stress Intensity Factor

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 (K1c) for each sample which is calculated by equation (1) and equation (2) the results (Table 3) (Figure 7). Figure 4 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 K1c 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_{1c} = 2.179 N.m$^{0.5}$, an increase of 286% compared to pure unsaturated polyester K_{1c} = 0.762 N.m$^{0.5}$. The addition of vinyl ester up to 70% increases the value of K1c 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_{1c} 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 (Ali et al., 2022; Farhad-Ali et al., 2022; Rahman et al., 2022; Abral et al., 2019).

Table 3 Stress Intensity Factor of UP/VE Blend at Room Temperature

Percentage UP - VE (%)	*Specimen*	K_{1c} (MPa.m$^{1/2}$)	Average	Standard deviation
100 – 0	1	0.434	0.762	0.285
	2	0.949		
	3	0.904		
70 – 30	1	1.383	1.566	0.584

	2	1.095		
	3	2.220		
30 – 70	1	2.045	2.179	0.133
	2	2.311		
	3	2.182		
0 – 100	1	0.969	1.277	0.527
	2	0.977		
	3	1.885		

4. 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_{1c} = 2.179 $N.mm^{0.5}$ and differs greatly in toughness from pure polyester whose value K_{1c} = 0.762 $N.mm^{0.5}$. The critical stress intensity factor increased from K_{1c} = 0.762 $N.mm^{0.5}$ to K_{1c} = 2.179 $N.mm^{0.5}$ (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.

Nomenclature

an	Initial crack length of	mm
B	Specimen thickness	mm
K_{1c}	Stress intensity factor critical	$Mpa.m^{0.5}$
P_Q	Load maximum displacement curve,	Newton
SG	Spesifik grafik	$gram/cm^3$
w	With specimens minus initial crack length	mm
wt %	Ratio mixed volume to volume neat UP	%

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Ethical issues

Not applicable.

Informed consent

Not applicable.

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Conflict of Interest

The author declares that there are no conflicts of interests.

Data and materials availability

All data associated with this study are present in the paper.

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