

Energy Absorption Behaviour of r-PET/Epoxy Laminated Composite with Varying Matrix Ratio in Quasi-Static Indentation Test

Asyran Muhammad Azman¹, Azrin Hani Abdul Rashid^{1*},
Nurul Zakiah Zamri Tan¹

¹ Department of Mechanical Engineering Technology, Faculty of Engineering Technology, Universiti Tun Hussein Onn Malaysia, 84600 Pagoh, Johor, MALAYSIA

*Corresponding Author Designation

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Abstract: The strip of recycled Polyethylene Terephthalate (r-PET) bottle waste used as a reinforcement in polymer composites for impact energy absorption application is little recognized. This study investigates the energy absorption behaviour of r-PET/Epoxy laminated composite with varying matrix ratios subjected to quasi-static indentation test. The r-PET bottle strip orientation was arranged to cross-ply structure to produce the laminated composite. Different epoxy and hardener ratio for the laminated composite was applied which are, 8:1, 7:1, 6:1, 5:1, and 4:1. The r-PET / Epoxy laminated composites were developed using hand-layup method. Grooveless r-PET bottles were selected to produce the r-PET strip and the strips were turned into cross-ply mats. The quasi-static indentation test was conducted to measure the maximum indentation force and total energy absorption of r-PET /Epoxy laminated composite. After the indentation test, the impact damage at front and rear surfaces was visually analysed. The findings show that different epoxy and hardener ratio affect the indentation performance of the r-PET / Epoxy laminated composite. This study evidenced the cross-ply r-PET / Epoxy laminated composite with the epoxy and hardener ratio of 6:1 has better indentation performance. The maximum indentation force and energy absorption were also improved drastically.

Keywords: Energy Absorption, Epoxy, Quasi-static Indentation, Recycled Polyethelene Terephthalate, Laminated Composite.

1. Introduction

PET is one of the most widely use polymers on the market. PET is widely used as a bottle. There are studies that exploit the strength of PET bottles for example in construction [1]. PET is one of such synthetic polymers with several applications due to its outstanding properties [2,3,4]. The possibility

*Corresponding author: azrin@uthm.edu.my

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of using recycled polyethylene terephthalate (r-PET) plastic bottles as a sustainable reinforcing material appears to be somewhat encouraging. Recycled Polyethylene Terephthalate (r-PET) has proven to have a great performance [5]. Studies on the treatment of r-PET laminated composite have been conducted such as studying the effect of layering sequence [6,7] and orientation of r-PET strip [8]. However, there are still many studies can be done to study the treatment of r-PET composite on other parameters such as the effect of the difference between the ratio of epoxy and hardener as a matrix [9]. This study is important to look at the composite r-PET behaviour on impact. The results of the study are very useful for design purposes and to identify the potential of composites for impact.

In order to sustain the structural load, strengthening and stiffness are provided via reinforcement where composites are not used alone but embedded in an organic matrix such as polyester and epoxy resins [10]. Even though polyester is easy to make, however previous study stated its disadvantages of polyester high shrinkage and only moderate mechanical properties [10]. Since epoxy is a lightweight resin with high mechanical qualities compared to other polymer matrices, epoxy is particularly useful for laminate composites because it can effectively saturate the reinforcer fibre [8]. Epoxy resins are a form of thermosetting synthetic polymer made up of two components: resin and hardener, according to prior research [11].

Epoxy resin has been widely used because of its excellent mechanical properties [10]. Still, the toughness of the cured epoxy resin is not satisfactory enough, and it is easy to crack again under low-temperature conditions. To increase the toughness of the epoxy resin, the hardener ratio is a critical parameter influencing the reprocessing ability of the thermosets that give elastic properties, combined with excellent mechanical properties and solvent resistance [1]. Minty et al., 2018 who studied single boron free E-glass fibres coated with either γ -aminopropyltriethoxysilane (APS) or glycidoxypropyltrimethoxysilane (GPS) by applying a matrix system consisting of Araldite 506 epoxy resin and triethylenetetramine as hardener [9]. In the study, thermomechanical properties such as interfacial shear strength, glass transition temperature, storage modulus and linear coefficient of thermal expansion. APS and GPS coated with system matrices having different ratios. The results of a study showed that the hardener-to-epoxy ratio can influence various thermomechanical properties related to determining the strength of the interface [9].

The 1:1 or 2:1 of epoxy and hardener is the performance baseline recommended by the manufacturer [12]. A different study has been examined in various ratios of hardener and epoxy. Sidharta et al., 2021 studies at a percentage of 3:2 where epoxy resin is slightly excessive, the modulus increases, and the elongation is shorter, indicating a brittle nature [12]. Zheng et al., 2021 study developed epoxy resin fracture toughness and self-healing efficiency increase with the increase in capsules content and reported the maximum healing efficiency of 80.00 % weight of epoxy [13]. This shows the high potential of the percentage used before epoxy in composites for future studies [14]. This shows the high potential of the percentage used before epoxy in composites for future studies [15]. In this study, fixing the same amount of epoxy with various hardener ratios has been conducted.

Besides, different many kinds of research using different materials in laminates composite through maximum indentation force and energy absorption [6]. Energy absorption is influenced by a variety of factors, including fibre type, matrix type, fibre architecture, specimen geometry, processing conditions, fibre volume percentage, and testing speed, according to Jacob et al., 2001 changes in these characteristics can lead to changes in composite materials' specific energy absorption [16]. When a material with a low modulus, energy absorption is increased [17]. Study from [6] investigated quasi-static indentation behaviour of unidirectional r-PET/Kevlar hybrid laminated composite through hand lay-up and vacuum bagging fabrication method where there is positive hybridisation of maximum indentation force and energy absorption of those two materials laminate composites. However, there are limited research on investigating the impact behaviour of r-PET composite [7]. Moreover, there are no research has been reported on exploring the effect of epoxy and hardener ratio of r-PET composite

towards the impact behaviour. This research aims to develop a better understanding of the impact response which are the maximum indentation force and energy absorption of r-PET/Epoxy laminated composite by varying the epoxy and hardener ratios. The r-PET/Epoxy laminated composite will be put under quasi static loading condition.

2. Materials and Methods

2.1 Materials

Groove-less mineral water bottle was used in this research. Epoxy was used as a matrix in this experiment. Epoxy function is to holds the r-PET mat in its designated position together in each layer and hardener as a curing agent. Figure 1 illustrates the groove-less bottle and the epoxy hardener used as the matrix reinforcement for r-PET/Epoxy laminated composite.

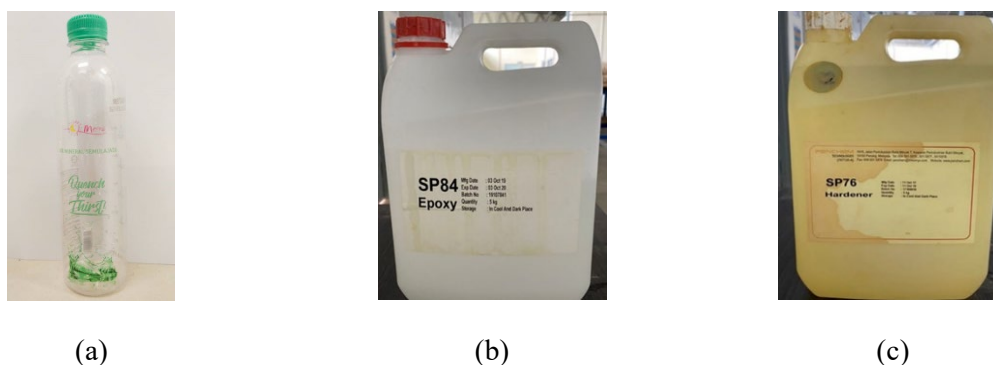


Figure 1: (a) Groove-less Mineral Water Bottle (b) Epoxy S84, and (c) SP76 Hardener

2.2 Methods

The bottle was cut into long continuous strip using special plastic bottle cutter and the width of the strips is 5 mm. Preliminary research has been conducted on various strip width (3 mm, 4 mm and 5 mm) and the results shows that 5 mm r-Pet strip exhibited the optimum tensile strength of 113.97 MPa and young's modulus of 2834.60 MPa. The fabrication of the unidirectional r-PET mat was accomplished by using hand weaving loom as shown in Figure 2 (a). Polyester yarn is used as the weft knot of the structure to ensure dimensional stability of the UD structure. The fabrication of the composite was executed using hand lay-up method as shown in Figure 2 (b).



Figure 2: (a) Weaving loom and (b) Hand lay-up process

The laminated composite is comprised of r-PET unidirectional mat with a matrix with epoxy resin SP84 and hardener SP76 as a curing agent. The primary purpose of the epoxy used in this analysis is to hold the r-PET string together in an ordered manner. The r-PET/Epoxy laminated composite was

arranged into four layers of $0^\circ/90^\circ/0^\circ/90^\circ$, which is cross-ply orientation in a single set as shown in Figure 3.

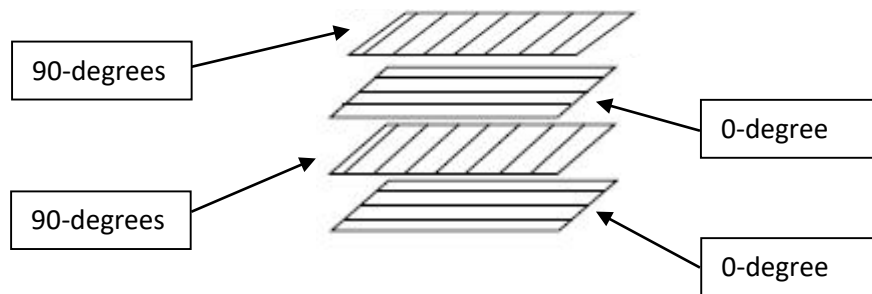


Figure 3: Laminated layers of r-PET/Epoxy in a single set

The laminated composite of r-PET/Epoxy was clamp before put in the oven with 30-degree Celsius in 24 hours. The reason why put into oven is because to maintain the temperature of the laminated composite. After 24 hours, the laminated composite of r-PET/Epoxy was cut into 100 mm x 100 mm size. Then, the r-PET mat physical properties in different ratios of matrix such as matt thickness weight and width were evaluated as shown in Table 1. Five sample configuration's thickness (t) was measured using the Vernier caliper and the mass of the r-PET mat sample was determined using a digital weighing scale. The sample was measured in 3 replications for each test.

Table 1: Physical properties of r-PET/Epoxy laminated composite

Sample Configuration	Weight (g)	Thickness (mm)	Density (g/mm ²)
cP4 (8:1)	24.44	2.70	3.35
cP4 (7:1)	25.81	2.74	3.44
cP4 (6:1)	28.94	2.75	3.83
cP4 (5:1)	29.96	2.78	3.88
cP4 (4:1)	32.85	2.80	4.19

R-PET laminated composite structure with ratio 4:1 displayed the weightiest, followed by 5:1, 6:1, 7:1, and 8:1. The reason of difference in weight of the r-PET laminated composite was caused by the amount of hardener used when fabricating the composite. Meanwhile, the difference in thickness was affected by the weight of the r-PET laminated composited. The weight and thickness displayed corresponding values with each other, where the higher the value of the weight, the higher the thickness of the composites.

Quasi-static indentation test was conducted on r-PET/Epoxy based composites with reference to ASTM D6264 using Universal Testing Machine (GOTECH). During the indentation test, maximum indentation force, energy absorption and specific energy absorption were measured. A quasi-static cross-head displacement rate of 1.27 mm/min was fixed throughout the indentation test. The laminated composite was clamped between the top and bottom support plates using four screws at the corners to avoid any slippage that affects the accuracy and reliability of the results during the indentation test. An indenter with a 12.7 mm diameter hemispherical tip was used to perform the indentation test. The set up was illustrated in Figure 3. The results were then represented by force-displacement curves to evaluate the energy absorption and maximum indentation force of the composite laminates. Finally, the damage behaviour of the composite laminates resulted from the indentation force were studied.

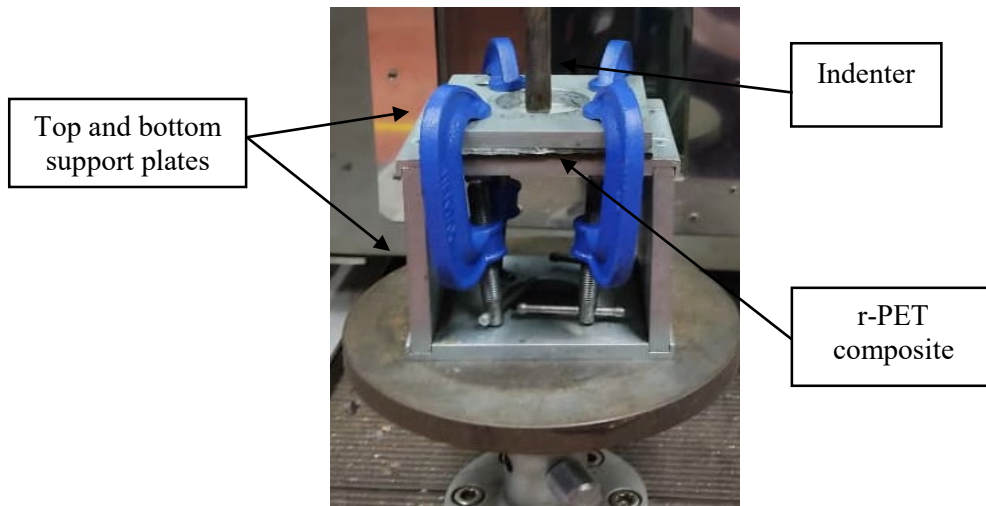


Figure 4: Setup of the quasi-static indentation test

3. Results and Discussion

3.1 Indentation Test

Findings obtained from the quasi-static indentation test are represented by the load-displacement curves to evaluate the energy absorption and maximum indentation force of the r-PET/Epoxy laminated composite represent in Figure 5. As can be seen in Figure 5, the load-displacement curves of the cP (6:1) and cP (7:1) showed a very similar trend irrespective of fibre matrix ratio configurations. The trend demonstrated the increase of indentation force along with the increase of displacement up to a maximum indentation force was reached. In fact, the indentation process of the composite laminates can be divided into three major regions. The load increased at the initial stage up to the peak point where the matrix cracking and initial delamination were noticed. After that, fibre breakage together with the higher extent of delamination occurred that resulted in the reduction in the load-carrying capacity of the composite laminates. Finally, the composite laminates were penetrated, that indicates the complete fracture, leading to the friction between the indenter and the composite laminates.

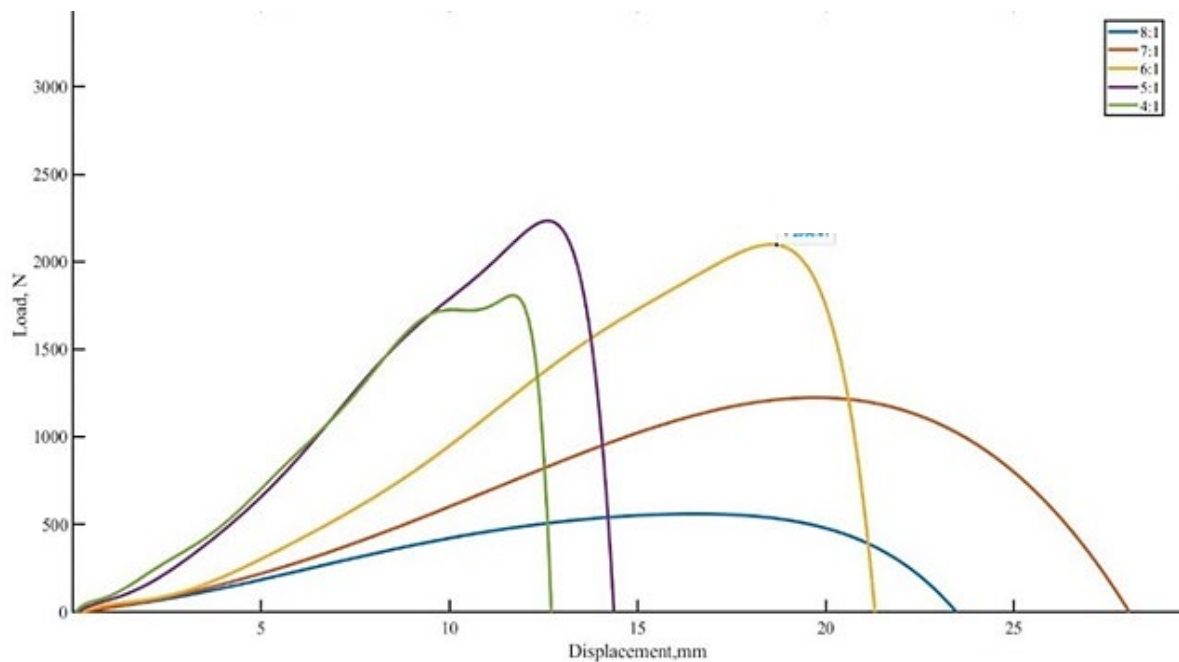


Figure 5: Load-Displacement curves of unidirectional r-PET / Epoxy composites

Based on the graph, the load-displacement curves can explain the elastic deformation of the composite laminate. The laminated composite in cP (6:1), cP (5:1), and cP (4:1) almost have similar curve pattern. At first stage, the laminated composite of cP (4:1) reached the maximum load first followed by cP (5:1) and (6:1). When started reaching second stage, the laminated composite started getting hard when continued reached peak value where significant drop of peak load in the load displacement curve was observed in composite laminated structure, and this sudden drop was due to the delamination that lead brittle in laminated composite structure. Meanwhile, after the load drop of cP (6:1), the load-displacement curve of cP (8:1) and (7:1) ratios continued to sustain the load but never exceeded the previous ratios peak load which has the reduction in the load-carrying capacity of the composite laminates. At this stage the penetration of laminated composites depends on its ductility. The laminate composite cP (6:1) had more advantages which consider one of the high peak load, small damage area, and low delamination. Even though cP (6:1) exhibited a brittle behaviour, but other laminates like cP (4:1) and cP (5:1) also has brittle behaviour that led to penetration, and delamination. Same goes to cP (7:1) and cP (8:1) laminate composite shows penetration and delamination on its ductility. The existence of brittle behaviour in the graph does not necessarily provide failure mode. Figure 6 also illustrated that laminated composite of cP (8:1) and (7:1) ratios had higher total displacement (more than 20 mm) compared to cP (5:1) and (4:1) ratios. While the maximum peak load for cP (5:1) was the highest, followed by cP (6:1) and cP (4:1).

The laminate composite cP4 (6:1) had higher total energy absorption because of its high strength compared to another laminate composite. The higher the ratio, the lower the strength of laminate composites but more flexible. This proves that the high ratio of cP4 (7:1) and cP4 (8:1) give more flexibility but low strength of laminate composites. The excessive number of hardeners makes the laminate composite is too flexible. Opposite with lower number ratio of hardener makes the laminate composite more rigid. This is because the laminate composite with fewer of hardeners depends on epoxy. According to a previous study Di Mauro et al., 2020 the hardener ratio is a crucial parameter influencing the reprocessing ability of the thermosets [19].

3.2 Energy Absorption

By measuring the area under the load-displacement curves, the energy absorption of the laminated composites was calculated. All the data were tabulated in Table 2, and the maximum indentation force and total energy absorption is illustrated in Figure 6. It was observed that cP4 (6:1) exhibited the highest maximum indentation force and total energy absorption, which are 17.14 N and 21.54 J, respectively. Meanwhile, cP4 (8:1) had the lowest maximum indentation force and total energy absorption by 5.21 N and 8.49 J. There is 11.93 N and 13.05 J different between the highest and lowest maximum indentation force and total energy absorption of cP4 (6:1) and cP4 (8:1). This indicates a rather large gap value for the two samples. However, the highest specific energy absorption was demonstrated by cP4 (7:1) with the value of 74.78 J/kg, while cP4 (8:1) shows the lowest specific energy absorption of 35.43 J/kg due to the lowest mass and density, as shown in Table 1 in chapter 2. Despite the difference in weight and density of the two samples of cP4 (8:1) and cP4 (7:1) quite close by 1.37g and 0.09g/mm² respectively, the indentation properties of total energy absorption play essential role to change the value of specific energy absorption. The greater the value of total energy absorption, the greater the value of specific energy absorption. However, even though the sample cP4 (6:1) show the highest total energy absorption, the weight and density of the cP4 (6:1) sample are relatively heavy compared to cP4 (7:1). There is difference of 3.13 g and 0.39 g/mm². This shows that the lighter the weight and density of the sample, as well as the higher the total energy absorption of the sample gives a high value to the specific energy absorption result [13,18]. The properties of the sample, such as penetration and delamination, are affected by the epoxy composition. Furthermore, the chemical composition of epoxy and hardener may play a significant role in why this occurs.

Table 2: Indentation properties of hybrid r-PET / Epoxy laminated composites

Sample configuration	Maximum Indentation Force (N)	Total Energy Absorption (J)	Specific Energy Absorption (J/kg)
cP4 (8:1)	5.21	8.49	35.43
cP4 (7:1)	11.83	19.11	74.78
cP4 (6:1)	17.14	21.54	71.18
cP4 (5:1)	13.04	15.75	53.33
cP4 (4:1)	11.87	12.10	38.31

Figure 6 shows the maximum indentation force and total energy absorption of r-PET/Epoxy laminated composite under quasi-static indentation. As we can see from the figure, bell-shaped histogram is shown. With a prominent mound/peak of cP4 (6:1) in the center and almost similar tapering to the left and right of other sample laminate composite with different ratios. The laminate composite of cP4 (6:1) show highest value for the maximum indentation force (more than 17 N) and the total energy absorption (more than 21 J). While cP4 (8:1) show the lowest maximum indentation force (less than 6 N) and total energy absorption (less than 10 J). The other laminate composite ratios are in the range between 10 until 20 for both maximum indentation force (N) and total energy absorption (J)

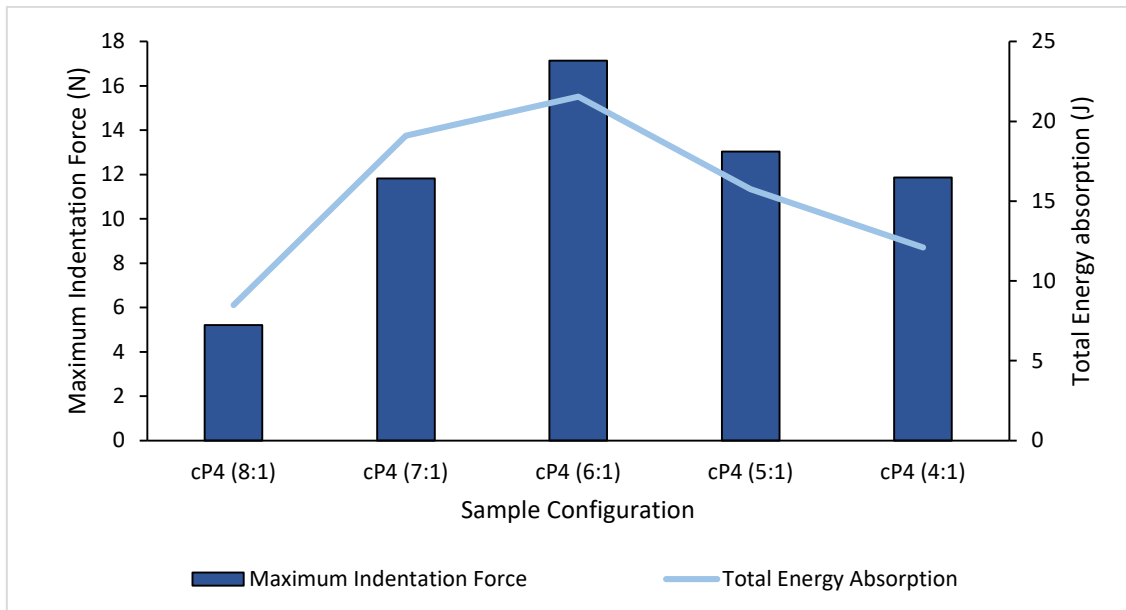


Figure 6: Maximum indentation force and total energy absorption of the hybrid r-PET/Epoxy laminated composite

Meanwhile, specific energy absorption of r-PET/Epoxy laminated composite under quasi-static indentation is shown in Figure 8. From the figure, a right-skewed histogram is shown. With a peak that is left of center of cP4 (7:1) and more gradual tapering to the right side of the graph of other sample laminate composite with different ratios. The laminate composite cP4 (7:1) had higher specific energy absorption that is more than 72 J/kg while cP4 (8:1) had the lower specific energy absorption which

less than 36 J/kg. The specific energy absorption for the other laminate composite ratios is in the range between 36 J/kg until 72 J/kg.

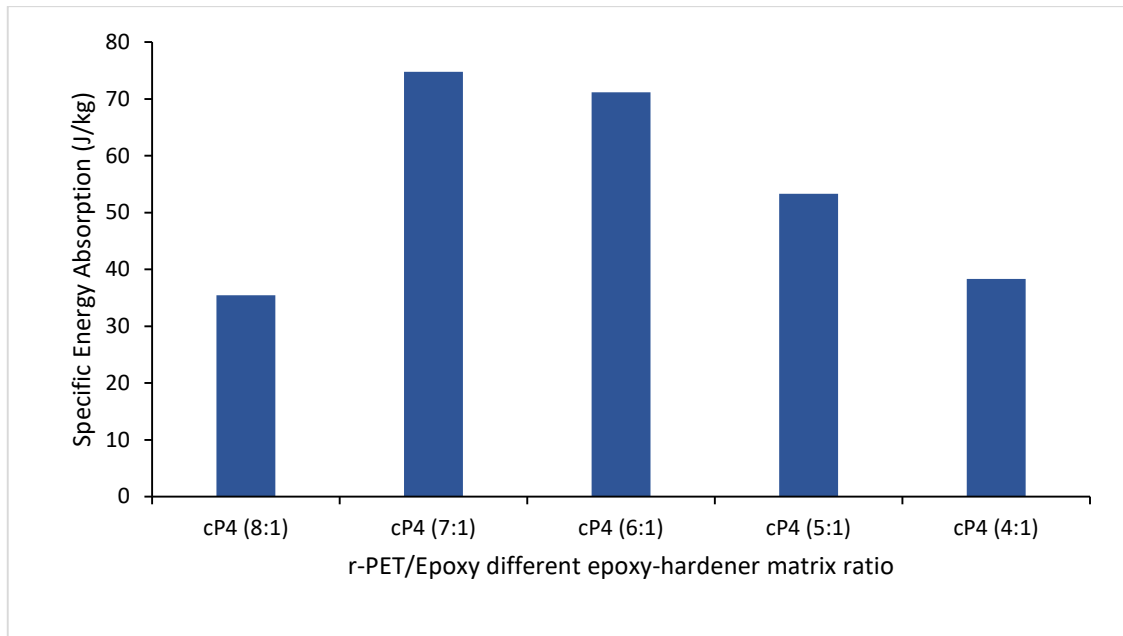


Figure 7: Specific energy absorption of r-PET/Epoxy laminated composite

3.3 Damage Assessment

The damage assessment after the indentation test was conducted on the r-PET/Epoxy laminated composite. The damage behaviours of the r-PET/Epoxy laminated composite were analysed on the front and rear surfaces of the fractured composites. It can be concluded that the damage behaviours of the laminated composite were highly influenced by the ratio of the epoxy and hardener. The damage behaviour that occurred during the test is depicted in Figure 8.

As can be seen in Table 3, the impact damage cP (5:1) based composite at the front was fully penetrated during the indentation test followed by cP (4:1), cP (7:1) and cP (8:1). Compared to the cP (6:1), cP (6:1) sample show great immune from penetration. Meanwhile, the impact damage at the back of the laminated composites also shows similar sequence preceded by cP (5:1) followed by cP (4:1), cP (7:1) and cP (8:1). This is attributed to the placement of high strength r-PET with a low matrix's ratio in the layers of the laminated composite, leading to the reduction in the crack propagation of the composite laminates. In addition, since this composite laminate contains epoxy, the surface tearing of epoxy is minimal and can be observed due to the delamination of the matrix from the surface reinforcement material.

This showed similar phenomenon to the previous studies; for example, study by Hussian Siyal, et al all explore about the mechanical strength and adhesiveness of hybrid laminates can be improved by using matrix materials with varied epoxy resin and epoxy hardener ratios [20]. glass fibre and Kevlar have very poor adhesion when 40 percent epoxy resin and 60 percent hardener are used. When compared to specimens made of 40 percent resin and 60 percent hardener in a matrix, the laminate with 60 percent epoxy resin and 40 percent hardener has superior mechanical properties, such as tensile strength hardness and minimum swelling ratio, whereas specimens made of 80 percent epoxy resin and 20 percent hardener have better bonding strength among the layers. Kevlar fibers reinforced with glass fibers have incredible capacity to produce less dense materials with superior strength, which attain high break and bear resistance characteristics. According to de Souza, et al adhesive material when used properly, can complement mechanical qualities, reduce component weight, increase durability, allow greater design flexibility, withstand high levels of stress, and increase composite strength [21].

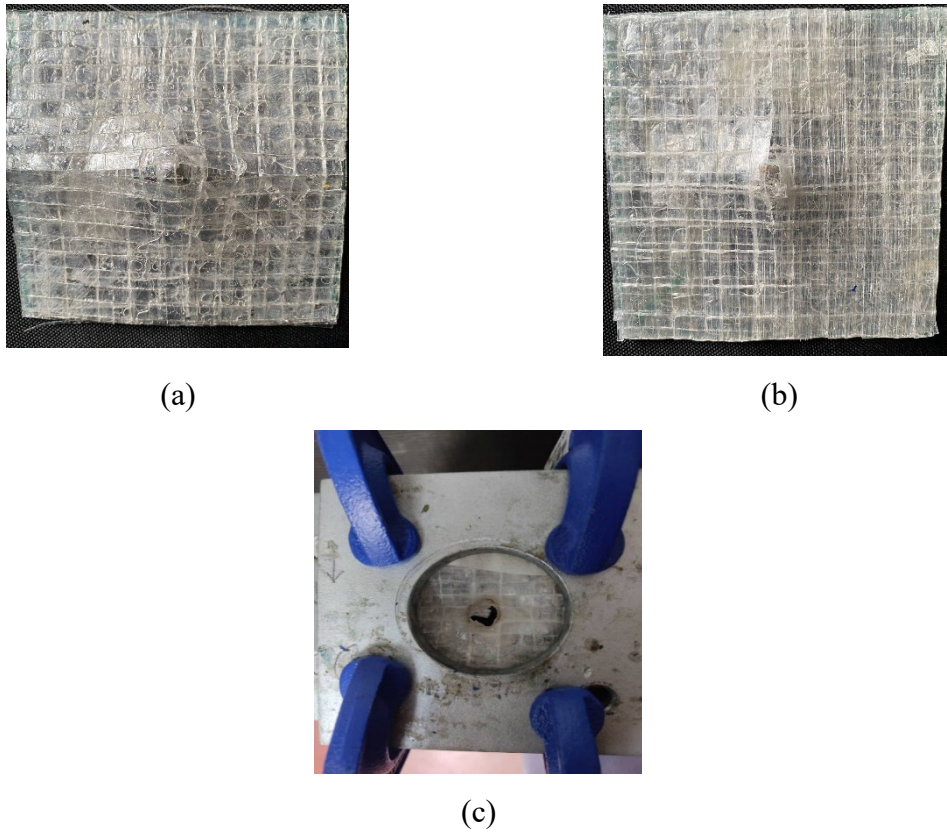
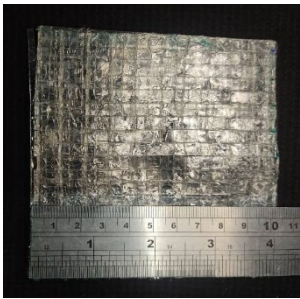
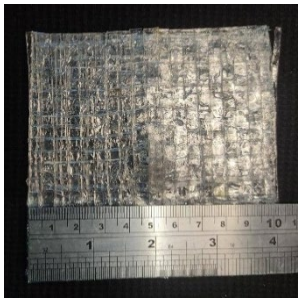
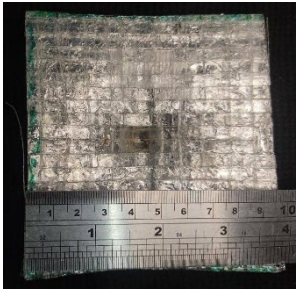
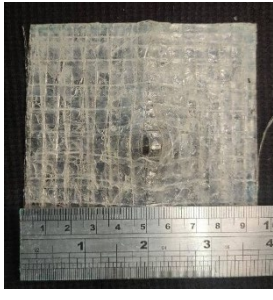
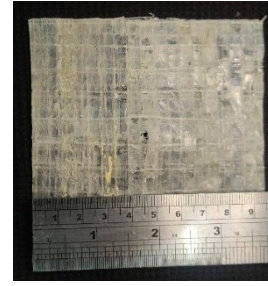
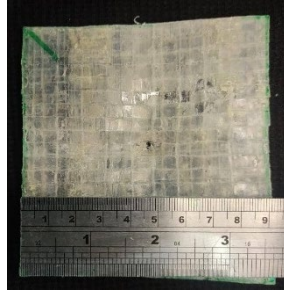
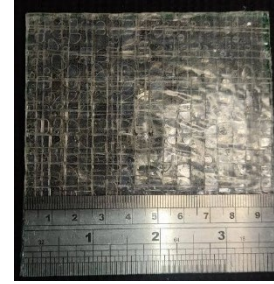
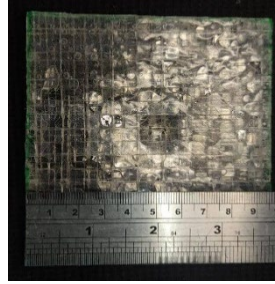
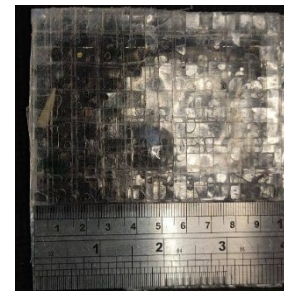
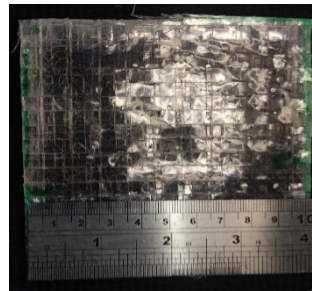


Figure 8: Damage behaviour; (a) Fibre tearing, (b) Matric cracking, (c) fully penetration

Table 3: Impact Damage (front and back) of r-PET/Epoxy laminated composite

Composite	Front	Back
cP (8:1)		
cP (7:1)		

cP (6:1)**cP (5:1)****cP (4:1)**

4. Conclusion

In conclusion, this research shows that the variation in epoxy-hardener ratio of r-PET/Epoxy laminated composite significantly influence the maximum load of indentation, energy absorption, and specific energy absorption. The different epoxy-hardener matrix ratio configuration of cP (6:1) exhibited higher energy absorption followed by cP (7:1), cP (5:1), cP (4:1), and cP (8:1) with a proportional relation to the maximum load of indentation. As for the specific energy absorption, cP (7:1) performed almost comparable with cP (6:1), where the values are 74.78 J/kg for cP (7:1) and 71.18 J/kg for cP (6:1). The findings proved that r-PET/Epoxy layers in cross-ply structure for cP (6:1) epoxy-hardener ratio had high potential to be used in composites for energy absorption applications. It can also be proposed that by applying the r-PET as a skin layer of the composite will provide a higher energy absorption and maximum load.

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