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Mechanical and Physical Properties of Glass-Fiber Reinforced Polymer Rods Manufactured by Different Pultrusion Methods

H. Alikhani [a], F. Sharifzadeh [b], H. Khoramishad [a]*

[a] School of Mechanical Engineering, Iran University of Science and Technology, Tehran, 16846, Iran
[b] Mechanical Department, Alzahra University, Tehran, 19938, Iran

KEYWORDS

Pultrusion
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Tensile test
Charpy test
Damage mechanisms

ABSTRACT

In this study, different methods of pultrusion including the out-of-mold and in-mold ultraviolet (UV)-cured and thermal pultrusion methods were investigated by comparing the mechanical and physical properties of the manufactured glass fiber-reinforced composite rods. The experimental results showed that the specimens fabricated by the UV-based methods were cured more uniformly and six times faster compared to the specimens made by thermal pultrusion. However, the specimens fabricated using the out-of-mold methods had higher diameter expansion and void content due to the lack of mold pressure during the curing process. Moreover, the mechanical responses of the specimens manufactured by different pultrusion methods were compared by conducting quasi-static tensile and low-velocity Charpy impact tests. The quasi-static tensile and Charpy impact strengths of the in-mold UV-cured specimens were 15.2% and 34.8% higher than those of the out-of-mold UV-cured specimens and 6.7% and 7.4% higher than those of the thermal-cured specimens, respectively. Furthermore, the fracture surfaces of the specimens were analyzed using SEM photography.

1. Introduction

The selection of appropriate composite manufacturing method has undeniable influence on the properties and quality of final products [1]. The pultrusion is one of the highly efficient processes suitable for continuous manufacturing of composite profiles reinforced with different kinds of fibers [2-3]. The applied tension to fibers during fabrication is an important parameter that causes higher mechanical properties for pultruded composite compared to the specimens fabricated by injection molding and filament winding methods [4]. The quality of final products fabricated using thermal pultrusion is considerably dependent on many parameters such as the pulling rate, the die temperature, the fiber content and resin viscosity that need to be controlled to achieve products with high quality [5]. Masuram and Roux [6] investigated the effect of fiber volume fraction on the pultrusion process. They reported that increasing fiber volume fraction on the pultrusion process led to fiber compaction in areas away from the mold wall subsequently reduced the wettability of fibers by making resin penetration more difficult. Moreover, the uniform dispersion of resin was disturbed in composites with high volume fraction. The resin content in central region was higher than near-wall region. Chen and Jia [7] employed the injection pultrusion process suitable for manufacturing the glass reinforced polyamide-6 composites with a high fiber content. They designed a specific chamber to improve the pultrusion speed and fiber impregnation. They reported that considering the heating zone temperature of 150°C resulted in highest flexural strength and flexural modulus, while increasing it to 180°C caused the highest inter-laminar shear strength for the composite. Grigoriev and Krasnovskii [8] found out that the friction force, which effects on pulling force, was dependent on pull speed, chemical shrinkage and resin pressure. The chemical shrinkage enhancement created residual stresses in the product which caused appearance of defects in composite.

Atrasia and Boukhili [9] investigated the temperature and curing degree inside the composite profile in different states of liquid, gel and solid. They found out that the pulling speed

* Corresponding author. Tel.: +98-21-77240540-50; Fax: +98-21-77240488.
Email address: Khoramishad@iust.ac.ir

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and pulling force effected the size and the position of different states. Santos et.al [10] optimized the die temperature using a swarm particle algorithm in order to reduce the energy consumption. The heat generated during the exothermic cure reaction was used as a heat source to enhance the die temperature. They reduced the energy consumption by 25% by optimizing the process. The consumed energy in pultrusion process was mostly related to the die temperature [11].

Curing of composites using ultraviolet (UV) irradiation is a curing method that has been used in recent years. This method can significantly reduce the resin curing time compared to the thermal process and consequently increase the efficiency of fabrication process [12-13]. The presence of photoniator in some polymeric resins such as vinylsters, polyesters and epoxies is activated by subjecting to UV light and can results in quick cure of composite matrices [14-16]. Endruweit et al. [17] found out that UV light can penetrate into composites through the voids and fiber bundles. Moreover, they reported that the light penetration capability depended on the thickness of fibers, the void size and fiber materials. Tina et al. [18] established a relation between the pulling speed, UV-light intensity and curing degree parameters in an out-of-mold UV-cured pultrusion process. They concluded that the irradiation intensity of 8 W/mm2 was sufficient for the fabrication of pultruded composites with the pulling speed of 2.62 mm/s. The out-of-mold curing of composites is a solution for reducing the fiber friction and pulling force in pultrusion process. However, the out-of-mold curing method cannot be used in thermal pultrusion and requires a fast-curing method, so using UV light is an appropriate option for out-of-mold curing of composites.

In this study, the properties of the glass fiber-reinforced composite rods manufactured by in-mold and out-of-mold UV-cured and thermal pultrusion were assessed. The curing degree, the diameter expansion, the void content, the surface quality and the mechanical properties were investigated experimentally. The tensile and low-velocity Charpy impact tests were carried out on the glass fiber-reinforced polymer (GFRP) specimens to determine the mechanical properties. Furthermore, the SEM fractography analyses were performed on the fracture surfaces of specimens to determine the micro failure mechanisms.

2. Materials and Methods

2.1. Materials

The GFRP rods with a diameter of 3 mm were fabricated using different pultrusion methods. The specimens were made of roving 2400 TEX glass fibers manufactured by Jiaxing Sunlong Industrial Co. China and the UV curable polyester resin supplied by Iran Polymer Co. Iran. Benzoyl peroxide (BPO)" was used in the thermal-curable polyester resin which was activated by high temperature and gave yellow color to the final product. 3 wt% of "BAPO-α aminoketone (combination of BAPO and 2-Dimethylamino-2-(4-methylbenzyl)-1-(4-morpholin-4-yl-phenyl)-butan-1-one (α aminoketone)" was used as photoinitiator of UV-curable resin which was activated by UV light and gave the crystalline color to FRP rod. The GFRP rods had the glass fiber total volume fraction of 70%.

2.2. Pultrusion processing

The schematic of pultrusion line employed in this study is shown in Fig.1. The fibers were impregnated with resin in a resin bath. Two tension rollers were used in the resin bath to guide the fibers and improve the wetting process. In order to remove the excess resin and pre-form the composite rod, a metallic tube with a length of 6 cm and a diameter of 3 mm was placed before the main die and UV chamber.

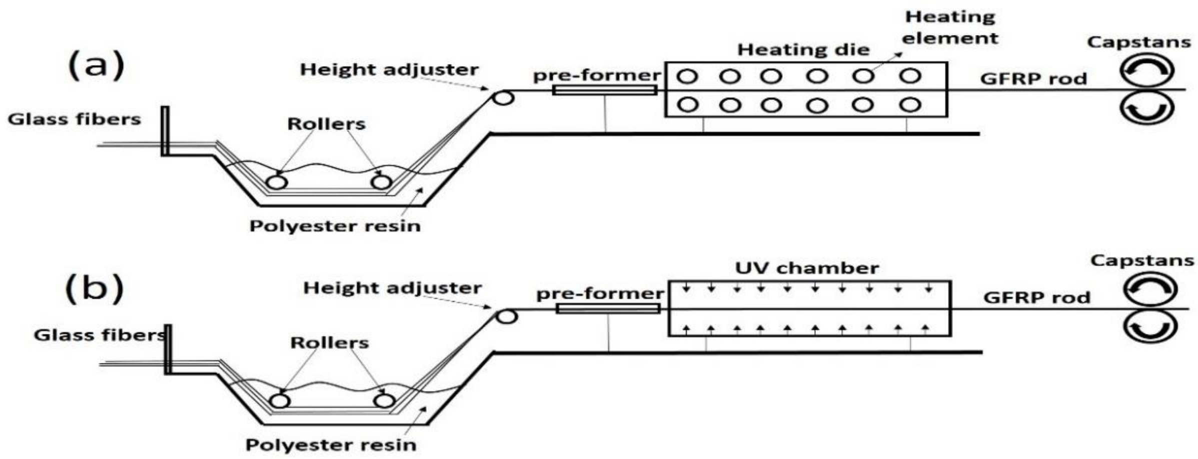

Fig. 1. Schematic of pultrusion lines (a) thermal-cure method (b) UV-cure method

In the thermal pultrusion method, the curing process was carried out in a metallic mold with a length of 800 mm heated by 600 Watt electric heating elements as shown in Fig. 2(a). The curing temperature of 150°C was adjusted uniformly in the metallic mold [7]. In order to control the temperature, two K-type thermocouples were embedded in the main mold. For the UV-cured pultrusion method, the curing process was performed in a UV chamber with a length of 300 mm equipped with 6-kW flood UV lamps with the wavelength of 365 nm and an aluminum reflector to increase the UV light centralization on the GFRP profile. For the in-mold UV cured pultrusion method, a transparent glass tube as the main die with the inner diameter of 3 mm and outer diameter of 6 mm was placed in the UV chamber.

3. Characterization of the GFRP composite rods

3.1 Curing speed

The time duration that GFRP composites require to be completely cured is a parameter restricting the speed of pultrusion line and production rate. Moreover, the curing degree of composites can considerably influence the properties of the manufactured product. Therefore, in order to have comparable samples manufactured by different pultrusion methods in terms of mechanical and physical properties, the UV-cured specimens were cured up to the same level of curing degree as the thermal-cured samples. The speed of thermal pultrusion production line was considered 400 mm/min and due to the heating die length of 800 mm, the curing time of 120 s was obtained for the thermal-cured glass fiber-reinforced polymer (GFRP-T) specimens. The speed of the out-of-mold and in-mold UV-cured pultrusion lines were determined such that to achieve the same curing degree for the out-of-mold-cured glass fiber-reinforced polymer (GFRP-O) and the in-mold glass fiber-reinforced polymer (GFRP-I) and the GFRP-T specimens.

It was shown in Ref [19] that there is a direct relation between the curing degree and electrical resistance of composites. Therefore, the curing degrees of the GFRP composite rods manufactured using three different pultrusion methods were measured by monitoring the electrical resistances of the specimens. Two copper electrodes with a diameter of 0.2 mm were embedded in the center of GFRP rod as the last cured region. The electrodes were placed at the beginning and end of GFRP rod and were connected to an ohmmeter to monitor the electrical resistance during the curing process. the measured electrical resistivity can be considered as volume resistivity, because it is the resistance to leakage current through the body of composite rod. The thermal and UV-cured pultrusion processes were simulated in static state and the electrical resistances of samples were measured. Therefore, the thermal-cured GFRP with a length of 800 mm was heated in heating die for 120 s, in order to absorb the same amount of heat as in the pultrusion line. The GFRP-O and GFRP-I samples with the length of 300 mm were subjected to UV light in static state until reaching the same curing degree of GFRP-T specimens. The variations of the electrical resistance versus the curing time for three different pultrusion processes are shown in Fig. 3.

The curing speed of the out-of-mold and in-mold UV-cure pultrusion methods were almost 6 times faster than that of the thermal-cured pultrusion method. Therefore, the GFRP-O and GFRP-I samples reached to the same curing degree of GFRP-T sample at 20.57 s and 27.24 s, respectively. According to the length of UV chamber and the obtained exposure times for the GFRP-O and GFRP-I samples, the speeds of the pultrusion line for the out-of-mold and in-mold UV-cured pultrusion processes were calculated as 87.46 m/min and 66.08 m/min, respectively.

The GFRP-O composite rods achieved the fastest curing speed indicating the higher UV-light permeability into the composite rods. In the out-of-mold method, the UV-light is completely absorbed by the specimen, while in the in-mold method, the mold was a barrier that absorbed a portion of UV-light and undermined the UV penetration power and consequently slightly reduced the curing speed of product.

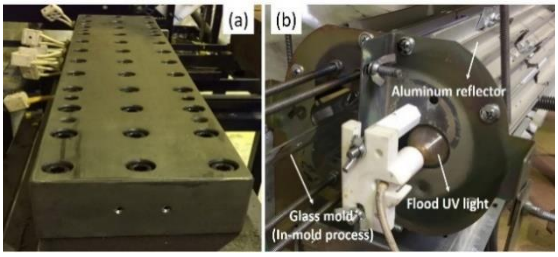

Fig. 2. (a) Heating die (b) UV chamber

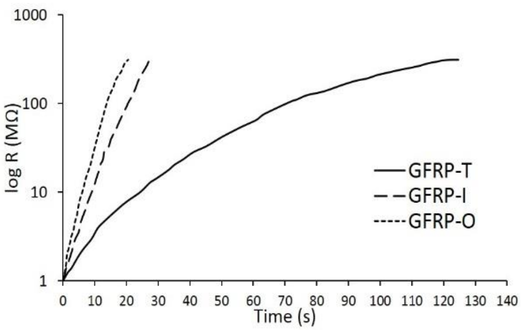

Fig. 3. The variations of the electrical resistance of fabricated GFRP composite rods versus the curing time

On the other hand, the UV-cure methods provide curing of composite rods by radiation heat transfer based on electromagnetic waves generated by UV-light with high permeability causing uniform curing degree across the cross-section, while in thermal pultrusion method, the curing process is conducted through conduction heat transfer that depends on the heat transfer coefficients of the fibers and resin and may not provide a uniform curing degree along the cross-section of the composite rods. The low thermal conductivities of the glass fibers and polymeric resin considerably lowered the conduction heat transfer rate than the radiation heat transfer rate in GFRP composite rods.

3.2. Diameter expansion

All The correlation between the expected (3 mm) and actual rod diameters is a parameter indicating the quality of the final product. The diameters of the GFRP rods fabricated by three different methods of pultrusion are compared in Fig. 4. As can be seen in Fig. 4, the diameter of GFRP rods manufactured by thermal and in-mold pultrusion methods were 2% and 1.33% lower than the expected diameter (3mm), respectively. This was because caused due to the inherent shrinkage of polyester resins during curing process giving rise to a small reduction in rod diameter [20]. Moreover, in the thermal pultrusion process, the appearance of a thin film of resin on the internal surface of heating die, shown in Fig. 5 (a), due to higher adhesion between the resin and metallic mold than resin and glass mold (in in-mold UV-cured method), led to further decrease in diameter of the manufactured GFRP-T samples than the GFRP-I samples. The diameter expansion of 2.3% was observed for the composite rod manufactured by the out-of-mold pultrusion method. This diameter expansion can be considered as one of the main drawbacks of the out-of-mold method. Due to the lack of mold pressure during the curing process on the samples manufactured by out-of-mold process, the glass fibers were free to move slightly outwards leading to increase in the product diameter after leaving the pre-former shown in Fig. 5(b).

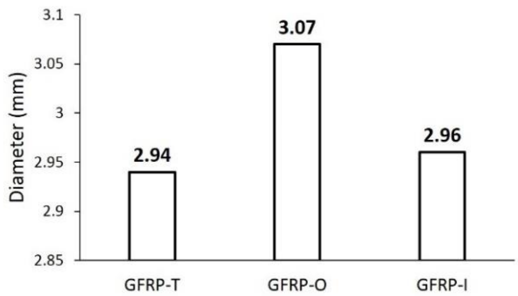

Fig. 4. Comparison between the diameters of the composite rods manufactured by different pultrusion methods

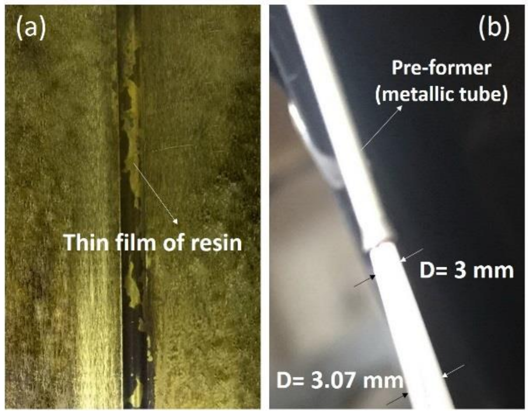

Fig. 5. (a) Thin layer of resin on the internal surface of heating die (b) Diameter expansion of GFRP after leaving pre-former in out-of-mold process

3.3. Void content in GFRP composites

Voids can act as stress riser and impose a negative effect on the mechanical properties of GFRP composites [21]. In order to compare the void formation in the samples manufactured by the three different pultrusion processes, the fiber, matrix and void contents were determined for the rods according to ASTM D3171-09 [22]. As can be seen in Fig. 6, the void content obtained for the samples fabricated by the out-of-mold pultrusion process was almost two times higher compared to the in-mold UV-cured and thermal methods. In the out-of-mold process, there was no mold pressure on the product during curing process giving rise to diameter expansion and gap formation between the glass fibers. The gaps between fibers were converted into voids. Therefore, the samples fabricated by the out-of-mold method were more prone to the void formation, while in the thermal and in-mold UV-cured pultrusion methods, because the curing process was performed under mold pressure, the gaps between the fibers was restricted and consequently the void formation was suppressed relative to the out-of-mold method.

The cross sections of GFRP samples were investigated by SEM micrographs as shown in Fig. 7. As can be seen in Fig. 7(a), there were plenty of voids in the GFRP-O specimen formed in the interface of resin and fibers, while fewer voids were observed in the central area of GFRP-T specimen because of the mold pressure applied on the specimen. Moreover, comparing the GFRP-T and GFRP-I samples revealed that the GFRP-I specimens had even lower void content compared to the GFRP-T specimens. This was because the high speed and uniform curing achieved in the in-mold UV-cured pultrusion method let to regular arrangement of glass fibers in the GFRP-I samples and higher final product quality with lower void content.

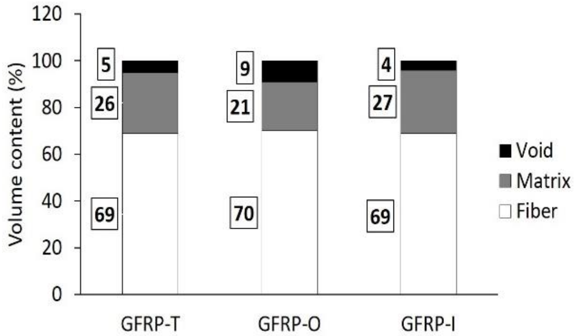

Fig. 6. The volume percentage of GFRP composite rods occupied by different constituents for the samples manufactured by different pultrusion methods

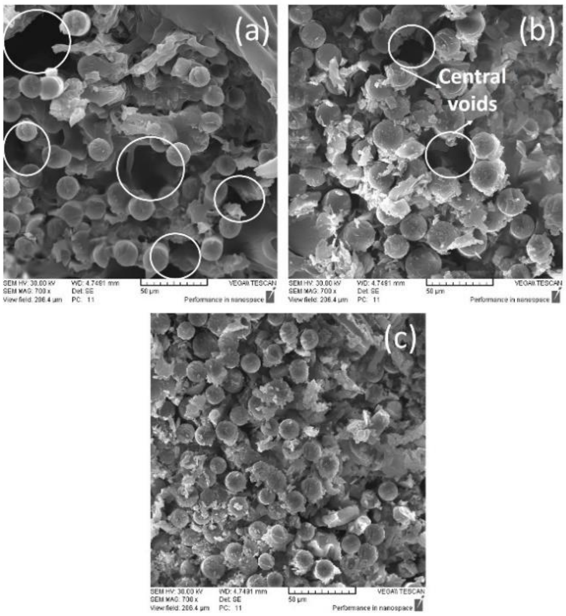

Fig. 7 The SEM micrographs captured from the cross sections of the pultruded profiles (a) GFRP-O (b) GFRP-T (c) GFRP-I

3.4. Surface quality

In order to investigate the effect of manufacturing method on the surface quality of GFRPs, the surface of samples was investigated by optical microscopy and SEM photography, shown in Fig. 8. As can be seen in Fig. 8(a), because of the lack of pressure in the out-of-mold curing method, the fibers were free to move outward to the specimen surface leading to a rough surface. Moreover, the appearance of voids on the surface of GFRP-O specimens was observable in the optical microscopy images as shown in more detail in SEM photographs. As can be seen in Fig. 8(b) and Fig. 8(c), for the GFRP-T and GFRP-I samples, the fibers were regularly arranged on the specimen surfaces and were covered by the resin providing a smooth surface with high quality. This is one the main advantages of in-mold curing of composites, as the glass fibers are compressed by the mold pressure. However, some small fragments detached from the polyester resin were observed on the surface of GFRP-T specimens which were the parts of resin film stuck to the surface of metallic mold shown in Fig. 5(a).

4. Mechanical properties of GFRP composite rods

4.1. Tensile properties

In order to determine the tensile properties, quasi-static tensile tests were carried out on the specimens according to standard ASTM-D3916 [23]. The tensile loading was applied on the samples with a loading rate of 2 mm/min and in order to assure repeatability of the results, each test was repeated at least 3 times. A 1000 kN Universal Testing Machine (UTM) was used for testing of specimens with length of 85 cm. An extensometer and a strain gauge were placed at the center of composite rod to measure the elongation during tensile tests. The top and bottom of composite rod were fixed by aluminum grip adaptors. The tensile strength, elasticity modulus, toughness and fracture strain were investigated and the results are shown in Fig. 9.

The GFRP-I specimens had the highest tensile properties. The tensile strength of the GFRP-I specimen was 15.2% and 6.7% and the elasticity modulus of GFRP-I was 7.3% and 1.7% higher than those of the GFRP-O and GFRP-T specimens, respectively. The high curing temperature of GFRP manufactured by thermal pultrusion process reduced the mechanical properties of GFRP-T compared to the GFRP-I specimens cured in low temperature.

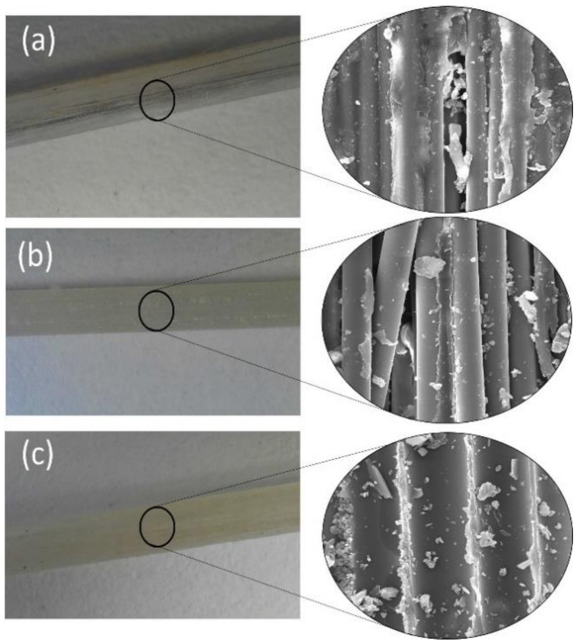

Fig. 8. The optical microscopy and SEM images captured from the surfaces of composite rods (a) GFRP-O (b) GFRP-T (c) GFRP-I

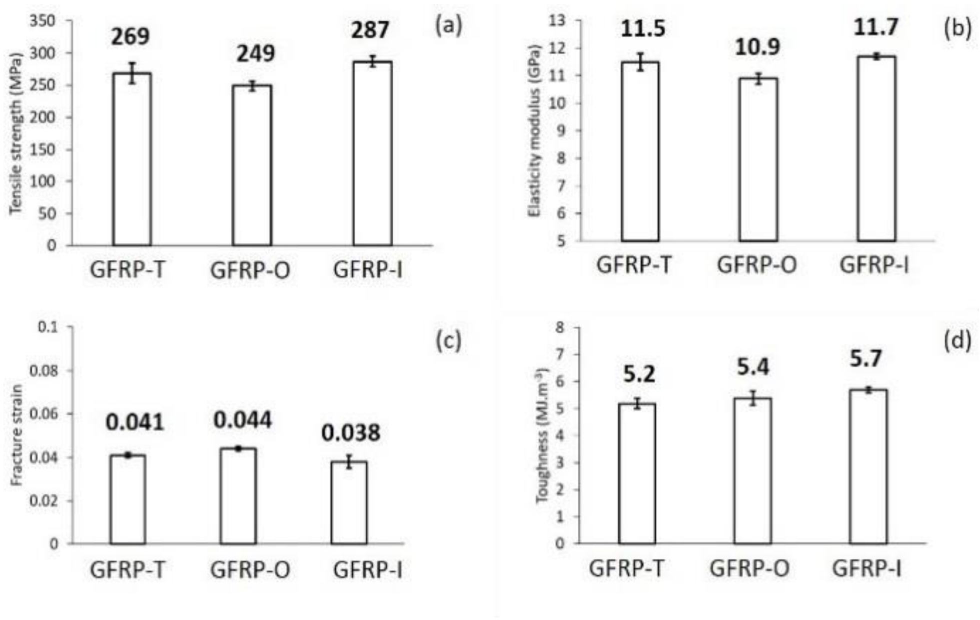

Fig. 9. The tensile properties of GFRP-T, GFRP-O and GFRP-I composite rods (a) Tensile strength (b) Elasticity modulus (c) Fracture strain and (d) Toughness

It is one of the main advantages of UV-cured pultrusion process that can preserve the mechanical properties of sensitive materials to high temperature through low-temperature curing. The high volume content of voids in the GFRP-O specimens was an effective factor on reduction of tensile properties in comparison with the GFRP-I specimens, even more effective than the high curing temperature applied in thermal pultrusion.

The GFRP-O specimens had more ductility relative to the other specimens. Toughness is a parameter indicating the energy absorption capability of materials and is measured as the area under the stress-strain curves. Similar to the tensile strength and elastic modulus, the in-mold UV-cured specimens provided the highest toughness compared to the other specimens.

The fracture surfaces of the samples subjected to tensile loading were investigated using SEM fractography. The presence of voids in the interfaces between the fibers and resin in the specimens manufactured by the out-of-mold methods caused weakening of fiber-matrix adhesion in GFRP-O samples. The reduction of fiber-matrix adhesion caused that fiber pull-out to be the dominant damage mechanism during tensile loading, as shown in Fig. 10(a). The matrix of manufactured samples was from the thermoset type of resin and incomplete curing of composite core caused necking of matrix due to higher plastic behavior and matrix elongation capability in the central region. Moreover, the reduction of matrix adhesion caused debonding fibers from the resin be more significant in the core of the GFRP-T composite rods. The uniform complete curing of the GFRP-I specimens and the mold pressure applied in the in-mold UV-cure pultrusion process suppressed the formation of voids and gaps between the fibers and increased the interfacial adhesion between the glass fibers and polyester matrix. As can be seen in Fig. 10(c), the glass fibers were fully embedded in the matrix and the fibers were covered with a thin and uniform layer of resin that can be considered as an evidence for the high surface quality of GFRP-I samples.

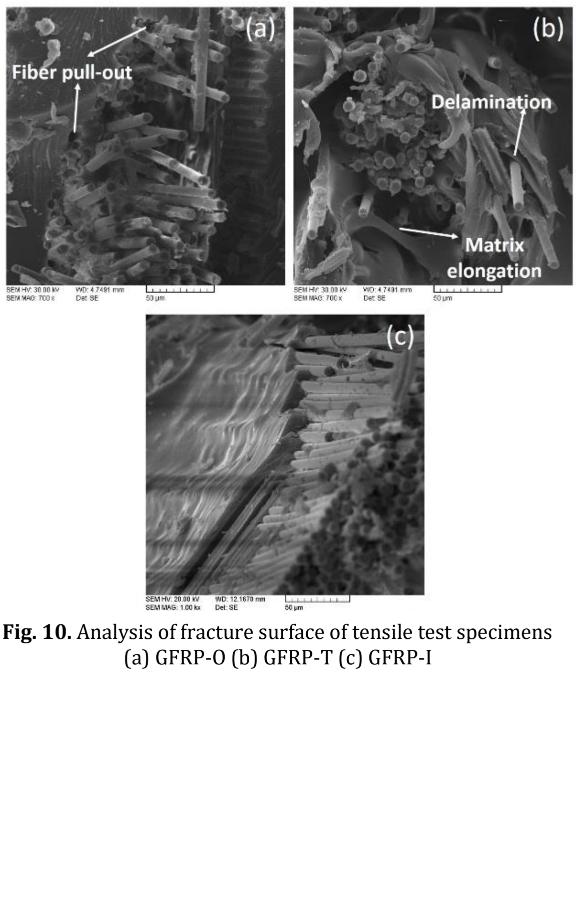

Fig. 10. Analysis of fracture surface of tensile test specimens (a) GFRP-O (b) GFRP-T (c) GFRP-I

4.2. Charpy impact test

Benefiting from a superior impact strength is one of the main advantages of glass fiber-reinforced composite structures. The relative flexible behavior of polymeric resins, that increases the plastic deformability and energy absorption capability, along with the high tensile strength of glass fibers and strong adhesion between the glass fibers and polyester resins can provide a structure with high resistance against impact loading [24-25].

In order to compare the impact properties of GFRP samples fabricated by different pultrusion processes, the Charpy impact tests were carried out according to standards ASTM D6110-10 [26] and ISO/EN 179-1 [27]. The span to diameter ratio of S/D=10 was considered for the impact test setup. Two different configurations of notched and un-notched specimens were tested by the impactor with the velocity of 6 m/s and the potential energy of 340 J. The impactor was a pendulum made of steel with angle tip of 45°. The schematics of notched and un-notched impact specimens are shown in Fig. 11. For the notched specimens, a small notch with a width of 0.8 mm and a depth of 1.2 mm was inserted in the middle of the span by a thin blade.

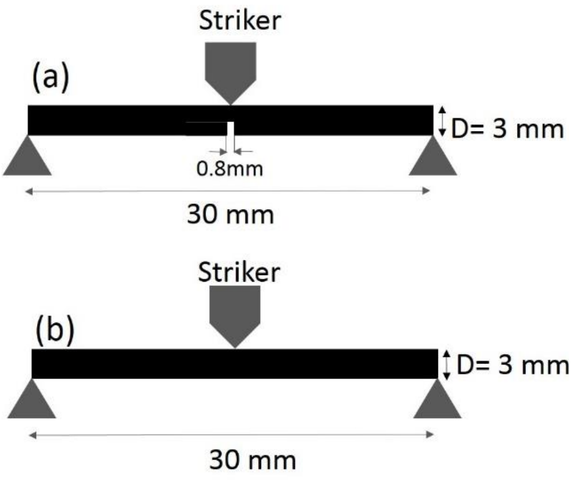

Fig. 11. Schematic of Charpy impact test configurations (a) notched (b) un-notched

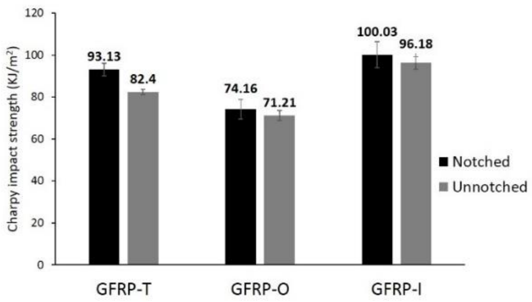

Fig. 12. Charpy impact strength of GFRPs manufactured by different pultrusion processes

The results of impact tests for both configurations are shown in Fig. 12. In all three cases, the Charpy impact strength of the notched specimens was higher than the un-notched configurations. This trend was reported in previous studies (e.g. [28]). The Charpy impact strength of the notched GFRP-I specimen was 34.8% and 7.4% higher than that of the notched GFRP-O and GFRP-T specimens, respectively, while for the un-notched specimens, the Charpy impact strength of the GFRP-I specimen was 35% and 16.7% higher than that of the GFRP-O and GFRP-T specimens, respectively.

Generally, the fiber-matrix interfacial adhesion has significant influence on the Charpy impact strength of composites [29-30]. Due to the higher fiber-matrix adhesion of the GFRP-I specimens compared to the GFRP-O and GFRP-T specimens, as can be seen from Fig. 10, higher impact energy was required for pulling out the fibers from the matrix in the GFRP-I specimens. For the notched specimens, the fiber-matrix interfacial failure (see Fig. 13(a)) and the fiber breakage (see Fig. 13(b)) were the dominant failure mechanisms around the notch tip. However, for the un-notched specimens, delamination was the failure mechanism which appeared in the form of crushing at the impact region of the striker, shown in Fig. 13(c).

5. Conclusions

The out-of-mold and in-mold UV-cured and thermal-cured pultrusion methods were employed to fabricate GFRP rods.

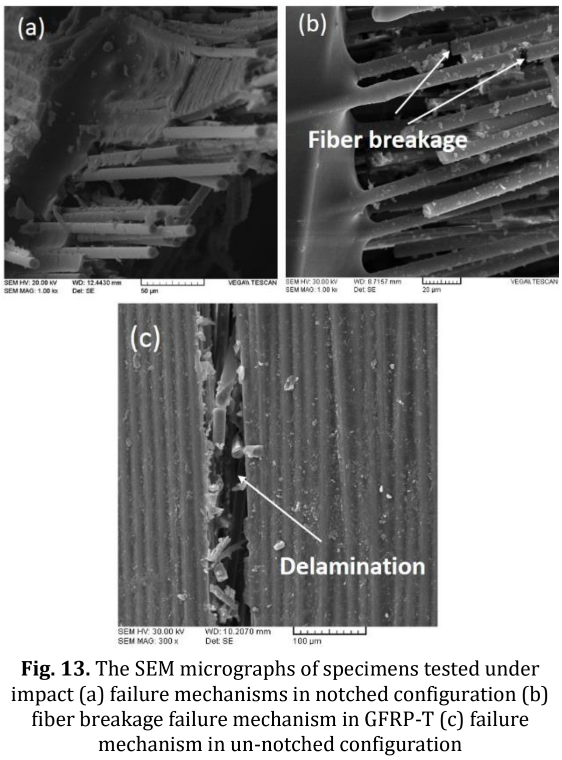

Fig. 13. The SEM micrographs of specimens tested under impact (a) failure mechanisms in notched configuration (b) fiber breakage failure mechanism in GFRP-T (c) failure mechanism in un-notched configuration

The curing speed, diameter expansion, void content, surface quality, quasi-static tensile and impact strengths were the considered parameters to compare the properties of the GFRP rods fabricated by different pultrusion methods. In out-of-mold process, the UV-light had high permeability into the composite rod which provided faster curing compared to the other methods. However, the lack of mold pressure during the curing process led to diameter expansion and void formation in the GFRP-O specimens, while in the GFRP-T and GFRP-I specimens the void content was lower with a value of less than 5%. The presence of voids at interfaces of fibers and matrix caused the fiber pull-out damage mechanism in the GFRP-O specimens and that gave rise to reduction in the tensile strength, elasticity modulus and Charpy impact strength, while the voids were minimized in the GFRP-I specimens resulted in highest tensile and impact strengths. Moreover, the matrix cracking was the dominant observed failure mode in the core of the GFRP-T specimens due to the longer curing time. The fiber breakage and fiber-matrix interfacial failure observed in the notched specimens and matrix cracking and delamination seen in the un-notched specimens were the dominant failure mechanisms. The curing speed of the UV-cured in-mold pultrusion was slightly lower than the out-of-mold method and the tensile, impact and surface properties of the GFRP-I specimens were higher than the GFRP-O specimens that caused this process be an appropriate option for manufacturing high quality GFRP composite with sensible amount of production rate. Because of the considerably lower curing speed of the thermal pultrusion, this method cannot compete with UV-cured pultrusion process commercially.

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