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EFFECT OF GLASS FIBER LENGTH AND THEIR CONTENT ON MECHANICAL AND THERMAL PROPERTIES OF FRP-FASTENERS

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ABSTRACT

A simple method for the preparation of FRP-composite fastenersconsisting of short and long length of glass fiber (GFS and GFL), unsaturated polyester resin (UPR), $CaCO_3$, MOS_2, and Zn-state is described. Short and long glass fibers were used as reinforce material. FRP-composite fasteners were prepared by first forming dough of polyester resin (UPR), fillers and internal and external lubricants with short and long glass fiberfollowed by compression molding and thermal curing ofpolyester resin. The FRP-composite fasteners were characterized by differential scanning calorimeter, thermo gravimetric analysis, universal testing machine and scanning electron microscopy. FRP-composite fasteners consists of 50 wt% of short length (3 mm) glass fiber and 50 wt% of long length (50 mm) glass fiberexhibited higher tensile strength (5.78 kN) than that of neat 100 wt% of short length glass fiber. Variation of glass fiber length in the FRP-composite fasteners did not affect much in the thermal properties. Mechanical properties of the composites mostly depended on the length of glass fibersand their contentin the composite. The FRP composite fastenerexhibited better mechanical properties as well as enhanced thermal stability.

Keywords: FRP-Fasteners, Unsaturated Polyester Resin, Glass Fiber, Mechanical Property, Thermal Property

I. INTRODUCTION

Conventional threaded fasteners in use are made of steel as they provide higher mechanical properties, easy availability and robust manufacturing process. These fasteners have limitations in applications like chemical process equipment, marine application, coastal areas where they can be subjected by chemical attack easily. Also some special application like cellular antenna mounts, computer testing facilities etc. Fiber reinforced plastic (FRP) fasteners can be effectively used in the following features such as corrosion resistance, ease of fabrication, low thermal conductivity, electromagnetic-frequency/ radio frequency transparency. Pultrusion is a commonly used process to produce FRP fasteners. In this process the fibrous reinforcements are allowed to pass through a resin bath of polyester/vinyl ester/epoxy which then enters into a pre-heated die and the complete polymerization takes place inside the die. The cured composite is continuously pulled by pullers and is then cut to the desired length. The nuts are separately fabricated. However the major disadvantage of pultrusion process

is that parts need to have a constant cross section like an extruded metal part. The parts are normally long but posses good strength in the longitudinal direction because of principal fiber orientation in this direction.

The qualitative aspect of a properly matured moulding was quantitatively expressed in terms of penetration depth, measured by a specially constructed softness indicator. The weight (wt.) % of CaCO3, Magnesium Oxide (MgO), Graphite (C) and Zinc Stearate (ZnSt having molecular formula [Zn ($C_{18}H_{35}O_2)_2$)]) along with the maturation time (T_m) were selected as process variables. Based on the ideal processibility conditions of SMC, the fiber orientation studies in threaded fasteners made out of FRP were carried out. These can offer high strength to weight ratio and ease in production and are useful in applications where corrosion resistance is a major design criterion. In this work, an attempt was made to orient the glass fibers according to the thread profile, along with the fiber orientation in longitudinal direction (like pultrusion) so as to impart the more strength to the component using compression moulding process. It was found that using compression moulding process, FRP bolts can be produced with proper flow of the matrix as well as fibers oriented in the desired direction according to the intricate geometry of the thread. This has resulted in good mechanical properties of FRP fasteners. Curing of resins has been a major thrust area of research for FRP products. An account of research in this area is given below:

A substantial decrease in curing time and hardness were seen when concentration of cobalt octoate and MEKP have been increased upto 0.02wt% and 2 wt% respectively [1].A comparative study of UP resin and unsaturated Vinyl ester (VE) resins based on the reaction kinetics of low temperature polymerization was performed [2].The curing behavior of the UP resin was studied by gel time and exothermic temperature measurements [3]. It was found that gel time was associated closely with initial rise in exothermic temperature. The findings revealed that the gel time increased from 10 minutes to 300 minutes when 4-tert butyl catechol was changed from 0 to 0.10 %, while it was reduced from 54 minutes to 16 minutes when 4-tert butyl catechol was varied from 0.05 to 0.8 %.Several disadvantages of conventional mixing of resin and curing agent before injection, like storing of hazardous polymerizing resins, wastage and cleaning have been highlighted [4].As a remedy, it was suggested that an on-line mixing method (similar to RTM process) in which curing agent mixes with resin when the later enters the mould could be employed. This would facilitate uniform gel time throughout the part by varying the mixing ratio of resin and catalyst. It has been claimed that gel time for the fabricated part was reduced by 20-25% by continuously varying the quantity of curing agent during injection. A semi-empirical model based on characterization of cure kinetics and resin viscosity for liquid composite moulding has been suggested [5]. Such models can be conveniently used for process prediction as well as optimization. To monitor the polyester cure monitoring, techniques like Fourier Transform Infrared (FT-IR) spectroscopy, DSC, rheometry and ultrasonic measurements have been used and the results have been correlated [6]. During the curing of UP/ styrene in presence of organically modified nanoclay, it was revealed that with the introduction 5wt. % of nanoclay, the tensile modulus and the fracture toughness were improved by 30% [7]. It was also claimed that the fall in the tensile strength can be avoided by adding $CaCO_3$ while achieving the highest value of fracture toughness. The researchers also developed a kinetic model based on free radical polymerization to predict the reaction rate as well as conversion profiles.Low-cost polymeric composites using untreated sugarcane fiber and polyester have been developed [8]. It was observed that the tensile strength of such composite was influenced more by fiber strength than the interfacial adhesion between the fiber and polyester. This resulted in significant improvements

in mechanical properties like tensile and compressive moduli as well as abrasion resistance.Several basic studies on the curing of UP resin highlighting the effect of inhibitors, initiators, accelerators, fillers and reinforcements on free radical polymerization can be found in handbooks on polyester resin curing and mouldingcompounds [9-12]. SMC is a ready to mould, fiber reinforced polymer matrix composite material which consists of thermosetting polyester resin, particulate filler and fiber reinforcement. SMC is very crucial for the production of FRP parts. As glass fiber reinforcement, in most cases cutglass roving's used. $CaCO_3$ is the most commonly used filler. MgO in the formulation can react with the free acid groups of the resin. This results after sufficient time, in an enormous increase of the viscosity of the SMC formulation. A leather-like sheet is then formed, which is almost non-sticky and easy to handle. At this stage, polymerization of resin does not take place.During the processing of SMC, the resin viscosity first decreases due to a higher temperature. The resin flows and fills the cavity. With increasing temperature; the cross-linking reaction rate rises to a maximum. Therefore the resin viscosity now grows. After the reaction the time depends on the size of the part and thenmould is cooled down to room temperature. Now, the part is removed from the mould.The origin of voids in SMC sheets and their transport has been investigated during the manufacturing steps like paste mixing, impregnation, thickening and moulding[13].It was found that the volume of entrapped air in the resin was around 18% and it increased further by addition of $CaCO_3$. The impregnation studies using Scanning Electron Microscope (SEM) have revealed a void content of 6.5% due to presence of fiber bundles.In a comparative study of carbon and glass fibers for SMC, it was also found that carbon fibers need slightly larger moulding forces (around 120 T) than glass fiber (around 100 T) for the same volume percent of reinforcement [14].The stiffness of SMC parts can be increased by provisions of ribs. However thematerial flow becomes complicated by introduction of ribs. As a result of change in orientation of glass fibers in such regions, the expected stiffness of the product can't be achieved. To find the material constant in a ribbed part, a method has been devised [15]. According to this method, the heterogeneity of the part wasexpressed as resin rich height by strength characteristics. The fiber orientation was characterized by FT-IR. The elastic modulus was calculated in the direction of average fiber orientation; however the method was giving errors for short rib heights.An experimental and numerical study was carried out to investigate the impact behavior of SMC plates [16]. Drop weight impact tests were carried out to examine the dissipated impact energies under different conditions of varying specimen thickness, initial impact velocity, and impactor mass. The highest dissipated impact energy of around 45 J was obtained for a 5 mm thickness. It was in the region of 12-136 J for impactor velocity of 3.2 to 4.5 m/s and has not shown any significant changes. It decreased from 14J to 10 J when impactor mass was increased from 2.2 to 4.5 Kg.Maleated hydroxylated soybean Oil (MHSO) and MaleatedAcrylatedEpoxidized Soybean Oil (MAESO) with styrene were successfully used for synthesis of SMC and BMC [17]. The flexural strength and moduli of these polymers varied from 61 to 87 MPa and 1.6 to 2.4 GPa, respectively. The tensile strength and moduli varied from 27 to 44 MPa and 1.6 to 2.5 GPa, respectively. These properties were comparable with those ofcommercially available UP resins commonly used in SMC applications.To characterize the flow of material and particularly of the glass fibers and their orientation in SMC produced by compression moulding different diagnostic techniques have been discussed [18].The details on SMC, its preparation and processing have been discussed at length in handbooks on moulding compounds [19, 20].

This study gives an account of experiments performed to find the right blend of SMC for the threaded fasteners. No such study has been carried out for producing threaded fasteners by compression moulding of SMC. The idea in using this process was to produce a FRP bolt to achieve a proper flow of the matrix as well as fiber orientation in the intricate geometry of the thread.

II. EXPRIMENTAL

2.1 Materials

The chemicals used were molybdenum disulphide (MOS_2), calcium carbonate ($CaCO_3$), Zn-sterate(Amrut Chemicals, India) andglass fiber (5mm and 15mm) supplied by BinnaniComposites, Goa, India,isophthalate unsaturated polyester resin (bis(1-(4-methoxy-4-oxobut-2-enoyloxy)propan-2-yl)isophthalate) diluted with styrene (UPR) (M_w=1500) (Mechanco Industries, India). The chemical composition of isophthalate unsaturatedpolyester resin was isophthalic anhydride, maleic anhydride, and propyleneglycol, bezoxyl peroxide (BPO) (98% Amrut Chemicals, India). Here,bezoxyl peroxide was used as an initiator.All chemicals were used as received.

2.2 Preparation of polyester-glass fiber composite fastener

Mixing: based on the combinations of the process variables for different experimental runs, different SMC compositions were prepared. In this work 50% resin and 50% glass fibers are used. In total glass fibers content the, 60% of the glass fibers are of long and remaining 40% are the short glass fibers. Initially a catalyst is produced by mixing the some quantity of resin with the benzyl peroxide. This acts as a catalyst. Then 8% of $CaCO_3$ is mixed in to it, stirred well then 6% of MgO, 5% of zinc sterite and 5% of molybdenum disulphide are used to produce a composite. $CaCO_3$ acts as a filler as well as thickener. MOS_2 gives good surface finish. Pressing: the dough has been carefully wrapped in polyethylene sheet and carefully passed through the rolling mill. This has produced a uniform SMC. Maturation: the SMC obtained is kept for maturation, to ensure consistency during experimentation; the maturation time was kept for 20 hours. The maturation quality was measured by softness. Loading of SMC: the properly matured SMC was weighted for each bolt to be approximately 25 gm. It is then pressed through of pre-form moulding die. This enabled the dough to take a regular shape resembling that of fasteners. It also facilitated easy loading of the pre-form. The three pre-forms were eventually loaded in the lower half of the three cavity of the mould. Pressing and heating of the pre-form: after loading the pre-form, the mould halves were just closed. The pneumatic actuator was then activated by the positional sensor, and it compressed the SMC from the front end in horizontal direction. Then the hydraulic pressure was raised to 200 bars. The moulds were heated to 200 ^{0}C to initiate cross linking of the resign and curing of the same took place. The process lasted till the temperature becomes 90 ^{0}C. Demoulding: upon completion of curing, the pneumatic cylinder was retracted and the mould halves were withdrawn. The bolts were ejected out. The flash as well as additional material adhered to with the bolts was cleaned.

Table 1. Different polyester-glass fiber composite fasteners compositions

Sample code	wt% ratio						
		Glass Fiber		Thickener		Lubricant	
	UPR	GFS	GFL	$CaCO_3$	MgO	MOS_2	ZnS
UPR-100/GFS-100/GFL00	50	26	00	8	6	5	5
UPR-100/GFS-80/GFL20	50	20.8	5.2	8	6	5	5
UPR-100/GFS-60/GFL40	50	15.6	10.4	8	6	5	5
UPR-100/GFS-50/GFL50	50	13	13	8	6	5	5
UPR-100/GFS-40/GFL60	50	10.4	15.6	8	6	5	5
UPR-100/GFS-20/GFL80	50	5.2	20.8	8	6	5	5
UPR-100/GFS-00/GFL100	50	00	26	8	6	5	5

Note: UPR-unsaturated isophthalic polyester resin (37% styrenated), GFS-short length (3 mm) glass fiber, GFL-long length (50 mm) glass fiber, ZnS- Zinc striate, MgO- Magnesium oxide, $CaCO_3$- Calcium carbonate.

2.3 Sample Characterization

Thermogravimetric analysis (TGA) and Differential scanning calorimetric (DSC) analysis were carried out for the neat polymers and composites by using DTG-60 and DSC-60 (Shimadzu, Japan), respectively. Thermal analysis was performed from 35 to 650 ^{0}C at a constant heating rate 10 ^{0}C/min in air atmosphere. The bolts were subsequently tested for maximum tensile load on a universal testingmachine (UTM) of 100 KN maximum capacity (FIE make, UTE-10 model). A specially designed fixture was used to fix the FRP nut-bolt assembly on the UTM. The load rate was 5 KN/min. The elongation rate was observed as 0.001 mm/min.The surface morphology of the composites was studied using a scanning electron microscope (SEM) (EVO 18, ZEISS, Germany).

III. RESULTS AND DISSCUSION

3.1 Thermal Analysis

DSC heating curves uncured polyester resin with benzoxyl peroxide (initiator), cured polyesterresin and polyester resin-glass fiber composite are shown in Figure1 (A). For uncured polyester resin with benzoxyl peroxide (BPO), the exothermic peak appeared at ~96^{0}C is a broad exothermic peak. The broadness of this peak is mainly due to the presence of higher heat of reaction(Figure1 (A) (a)), which corresponds to the thermally activated free radical polymerization of polyester resin in presence of benzoxyl peroxide [21]. In case of cured neat polyester resin and cured polyester resin-glass fiber composite (Figure 1(A) (b) and (c)) the exothermic free radical polymerization peak of polyester resin was absent (Figure1(A) (b) and (c)). This indicates that, polyester resin were fully polymerized bycross linked with styrene (co-monomer) during composite preparation as the moldingand curing was performed at 150 ^{0}C. Moreover, the thermal stability of cured polyester-glass fiber composite was also performed by DSC and compared with neat cured polyester resin (Figure 1(B) (a) and (b)). In case of neat cured polyester resin (Figure 1(B) (a)), an exothermic peak at ~352^{0}C and 498^{0}C wereobserved, which was due to the thermal-oxidative decomposition of polyester resin [22]. However, in case of cured polyester-glass fiber composite,an exothermic peak observe at 377^{0}C and 528 ^{0}C. These results indicate that, the thermal stability of composites has higher than the neat polyester resin. This may be due the absorption of heat

at glass fiber and lead to delay the thermal degradation of polymeric matrix of composite, as comparative to neat polyester resin.This result indicates that, prepared composite material has the more thermal stability than neat polyester resin.

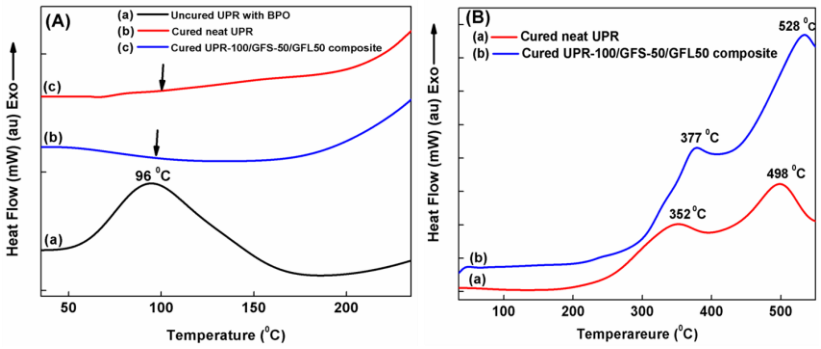

Figure1.(A)DSC thermogram of (a) uncured UPR with BPO, (b) cured neat UPR, (c) cured UPR-100/GFS-50/GFL50 and(B)DSC thermogram of (a)cured neat UPR, (b) curedUPR-100/GFS-50/GFL50

TGA thermograms of polyester resin and polyester-glass fiber composite are shown in Figure2. These thermograms revealed that, the thermal stability of the composite was found to be more than pure polyester resin. The degradation of all the composites was found in the temperature range of 350-480 ^{0}C. The char yield at 650 ^{0}C of the composite was found higher in polyester-glass fiber composite. From TGA of neatpolyester and their glass fiber composites, temperature at 5% weight loss ($T_{5\%}$), temperature at 10% weight loss ($T_{10\%}$) and char yield (%) at 650 ^{0}C in air were determined and listed in Table 2. It was observed that for all composition of composite, the temperature at 5 and 10% weight loss was found to be higher than the neat cure polyester resin. This may be duepresence of inert and thermally stable glass fiber in composites enhances the overall thermal stability of the composites. Moreover, from the TGA thermogramof composite, it was found that the amount of residual weight at 650^{0}C of the polyester-glass fiber composites was close to the actual amount of added wt% glass fiber in the composites. That may

be due to the high thermal stability of glass fiber, thus glass fiber was not experienced any weight loss in the temperatures range of 30-650 ^{0}C.However,polyester resinhas been completely decomposed at same temperature. This indicates that, the amount of char in each composite compositioncorresponds to the amount of glass fiber present in the composites. Moreover, there is no anysignificant effect of length of glass fiber on the thermal stability of composite.

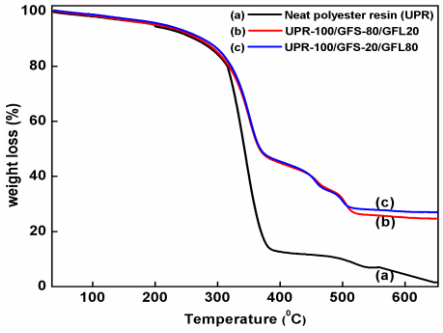

Figure 2. TGA thermograms of (a) neat polyester resin (UPR), (b) UPR-100/GFS 80/GFL20 composite, (c) UPR-100/GFS-20/GFL80 composite.

Table 2. Temperatures required for 5% and 10% weight loss of the composites due to thermal decomposition in air.

Sample code	T 5% (^{0}C)	T 10% (^{0}C)	Char Yield (%) At 650 ^{0}C (approx)
Neat polyester resin (UPR)	197	259	1
UPR-100/GFS-80/GFL20	205	268	24
UPR-100/GFS-50/GFL50	210	269	25
UPR-100/GFS-20/GFL80	207	266	24

3.2 Mechanical Properties of Composite

Tensile strength and modulus were performed to determine the mechanical properties of the polyester-glass fiber composite fasteners (Figure 3) and results are tabulated in Table 3. In our previous study we have observed that, presence of an optimum amount of 8 wt% MOS_2, 5wt% $CaCO_3$, and 2 wt% Zn sterate, significantly enhances the mechanical properties of composites. To determine the optimum amount of loading level of long glass fiber, required to achieve good mechanical property, samples were prepared by blending 4 mm (short glass fiber) and 10 mm (long glass fiber) length glass fiberwith variation of 10 to 90 wt% ratioin polymeric matrix and tensile tests were performed. It was observed that tensile strength and modulus of the composites increased with increasing amount of long glass fiber (up to 50 wt%) and then started to decrease (Figure 3). In case ofshort glass fibers are placed at the curves threads during processes and resulted in, increase the strength of thread. Therefore, loading stress was easily transferred from polymer matrix to short glass fiber which is placed in thread curve and ultimately tensile strength and modulus of the composites increased, however the short length glass fiber fastenersfound the failure at neck. On the other hand, a fastener with longer glass fiber has better tensile property but they failed at threads. This could be due to the fact that longer glass fibers could not able to get oriented according to thread profile. However, the 50 wt% of long and 50wt% short combination of glass fibers found to give better load bearing capacity at neck as well as at threads of fasteners, this might be due to the, long glass fiber easily oriented at the core of fasteners and lead to result in better loading bearing capacity at the neck, while short length glass fiber oriented according to thread shape and produce well defined threads without any failure. Therefore, loading stress was easily transferred from polymer matrix to long glass fiber at neck core and at short glass fiber at threads and this ultimately leads to give better tensile strength and modulus to FRP-fasteners. Figure 5 demonstrated the fractured fastenersUPR-100/GFS-50/GFL50 composite.

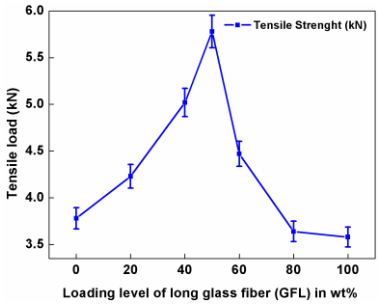

Figure3.Relation between loading level of long glass fiber and mechanical properties of the composites.

Table3.Mechanical properties of the composites with the variation of short length glass fiberand long length glass fiber in the composite.

Sample code	Tensile load(kN)	Mode of failure under tensile load
UPR-100/GFS-100/GFL00	3.58	Neck failure
UPR-100/GFS-80/GFL20	3.64	Neck failure
UPR-100/GFS-60/GFL40	4.47	Neck failure
UPR-100/GFS-50/GFL50	5.78	Thread shear
UPR-100/GFS-40/GFL60	5.62	Thread shear
UPR-100/GFS-20/GFL80	4.23	Thread shear
UPR-100/GFS-00/GFL100	3.78	Thread shear

3.3 SEM Analysis of Composite

The SEM photomicrographs of the fractured surfacesof the neat blend (without fumed silica) and fumed silica field compositesare shown in Figure4(a) - (e).It was observed that, there was continuous and homogonous matrix (neat UPR) (Figure 4(a)). However, in case of composites,whitecircular of glass fiber indicate the position of the dispersed glass fiber in the polymeric matrix. In case of UPR-100/GFS-100/GFL00 composite(Figure 4 (b))and UPR-100/GFS-80/GFL20composite (Figure 4(c)), it was observe that, homogenous dispersion of short glass fiber in polymeric matrix with less anisotropy. In case of UPR-100/GFS-60/GFL40 composite andUPR-100/GFS-50/GFL50 composite(Figure 4 (d) and (e)), it was observed that long and short glass fiber dispersed continuously and with less anisotropy in polymeric matrix.In contrast, in case of UPR-100/GFS-40/GFL60 composite (Figure 4 (f)), and UPR-100/GFS-80/GFL20composite (Figure 4(g)), it was clearly observed that, long glass fiber are agglomerated and irregular in distribution and has larger anisotropy in polymeric matrix as compared to the UPR-100/GFS-60/GFL40 composite (Figure 4 (f)) and UPR-100/GFS-50/GFL50composite (Figure 4 (g))and this could leads to decrease the overall mechanical properties of the composite. In case of UPR-100/GFS-00/GFL100 composite (Figure 4 (h)), it can clearly observed that the long glass fiber orient in unidirectional without any short length glass fiber.

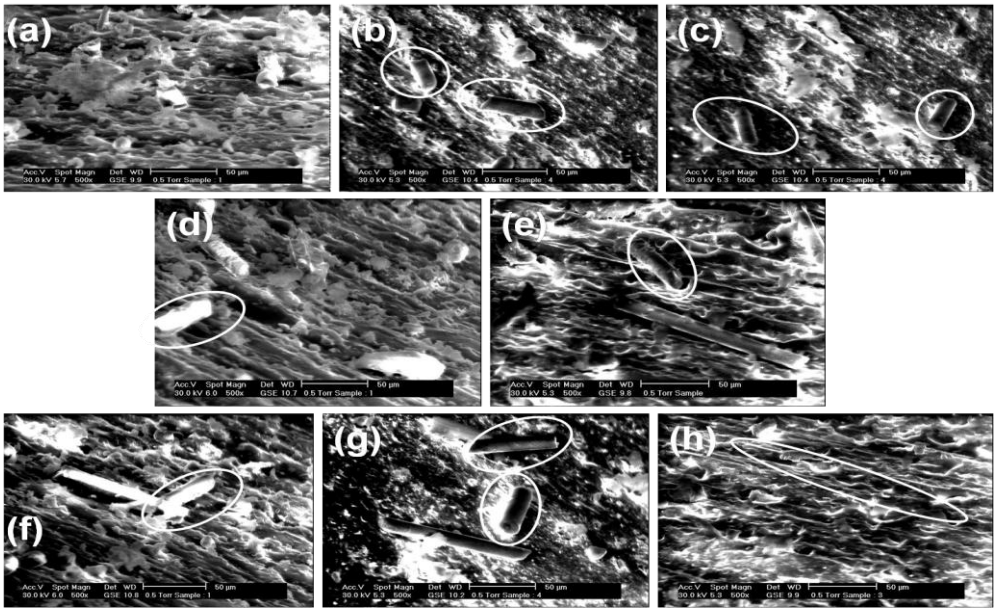

Figure 4. SEM photomicrograph of (a) UPR-100/GFS-00/GFL00 matrix, (b) UPR-100/GFS-100/GFL00 composite, (c) UPR-100/GFS-80/GFL20 composite, (d) UPR-100/GFS-60/GFL40 composite, (e) UPR-100/GFS-50/GFL50composite, (f) UPR-100/GFS-40/GFL60 composite, (g) UPR-100/GFS-20/GFL80 composite, (h) UPR-100/GFS-00/GFL100 composite

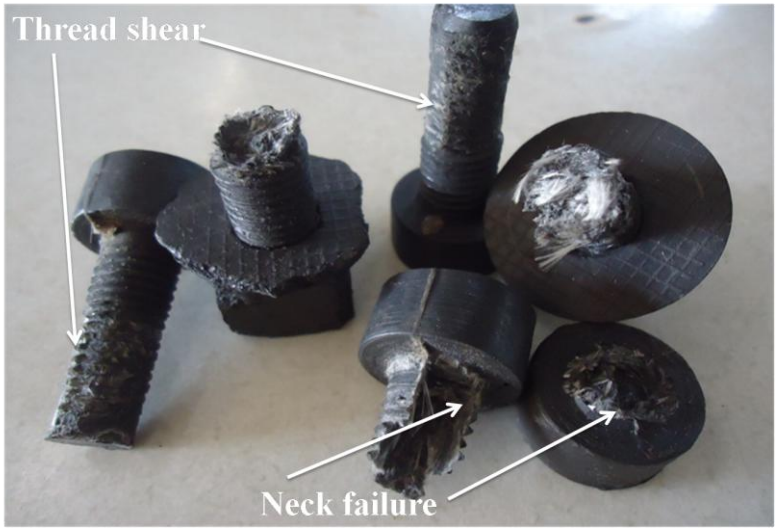

Figure 5.UPR-100/GFS-50/GFL50composite fasteners (M14 x 2) size showing the fracture under the tensile load.

IV. CONCLUSION

Polyester-glass fiber based fasteners were prepared by compoundingunsaturated polyesterresin with different length of glass fiber in presence of $CaCO_3$ (filler), MOS_2 (external lubricant) and Zn striate (internal lubricant) by employing a simple compression molding method. Thermal stability of the composites was found to be increase as compared to the neat UPR resin. Prepared composite has the better thermal stability than pure UPR. Tensile strength and modulus of prepared composites revealed that 50wt%is the optimum amount of long glass

fiber that can be used in composite preparation. This method is very simple and readily availableUPR was used to make fasteners with different length ofglass fibers. So, these FRP-composites fasteners can easily be prepared in elevated scale. As the prepared FRP-fastenerscomposites possess good thermal stability along with superiormechanical properties, these composites have the potential to be used in union of complex structures where high mechanical strength and thermal stability subjected.

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List of Table captions

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Table2. Temperatures required for 5% and 10% weight loss of the composites due to thermal decomposition in air.

Table3. Mechanical properties of the composites with the variation of fumed silica in the composite.

List of Figure captions

Figure1. DSC thermogram of **(A)**(a) uncured UPR with BPO, (b) cured neat UPR, (c) cured UPR-100/GFS-50/GFL50 and, **(B)**(a)cured neat UPR, (b) curedUPR-100/GFS-50/GFL50

Figure2. TGA thermograms of (a) neat polyester resin (UPR), (b) UPR-100/GFS-80/GFL20composite, (c) UPR-100/GFS-20/GFL80composite.

Figure3. Relation between loading level of long glass fiber and mechanical properties of the composites.

Figure 4. SEM photomicrograph of (a) UPR-100/GFS-00/GFL00 matrix, (b) UPR-100/GFS-100/GFL00 composite, (c) UPR-100/GFS-80/GFL20 composite, (d) UPR-100/GFS-60/GFL40 composite, (e) UPR-100/GFS-50/GFL50composite, (f) UPR-100/GFS-40/GFL60 composite, (g) UPR-100/GFS-20/GFL80 composite, (h) UPR-100/GFS-00/GFL100 composite.

Figure 5.UPR-100/GFS-50/GFL50composite fastener (85 mm x 12 mm x 1 mm) showing the fracture under the tensile load.