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Synergistic improvement of epoxy derived polymer composites reinforced with Graphene Oxide (GO) plus Titanium di oxide(TiO_2)

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ABSTRACT

This technical article top up in presenting the research over composites consisting of Graphene oxide(GO), Titanium di oxide (TiO_2), epoxy resins along with a known curing agent. Therefore, common influences of GO as well TiO_2 substances upon the curing process of epoxy systems all along with thermo-mechanical performances of GO-TiO_2 epoxy composites were conversed in detail. Herein, assimilation of GO particles resulted to accelerate the curing process of epoxy systems. GO-TiO_2 epoxy composites were found to be capable of increasing ultimate tensile strength by up to 59%, flexural strength by 79%, and notable increment in their mechanical properties too was evident while compared with the outcomes of unmodified epoxy based thermosets developed for the purpose. Fracture surface along with tensile properties were made experiential by the way of field-emission SEM and further observations over these micrographs shows in for the occurrence of ductile fracture mechanism with respect to GO addition. Thermal analysis results make public that decomposition temperature of developed composite was up to 470 °C.

1. Introduction

In current lieu, composite materials find extensive applications in structural components due to its better mechanical properties and amended fatigue life. Among them, high performance polymer based composite materials achieves wider range of application under hard adverse working environments. For that conditions, the selected materials must deliver unique thermal and mechanical properties with combination of low specific weight and high degradation resistance in order to ensure economic efficiency and safety factors [1–4]. In this, thermoset based polymer matrix, namely epoxy resins has already covered some demanded properties that finds in major applications such as for adhesive joint, surface coating, transport industry and some other structural engineering application of similitude. Epoxy resins deliver certain unique properties viz. easy to process under different conditions, better thermal and mechanical properties [5–10] besides possessing higher number of cross linked networks that leads to high rigidity and strength as well. However, its wider range of potential applications was limited owing to such other presence of networks which in turn tends to increase the brittleness [11–13]. This fact made the researchers worldwide heading up into such activities to concentrate on improvising the toughness of epoxy based resin. Addition of filler materials into the epoxy resin was considered to be one of the most efficient methods for property enhancement such as better stiffness and strength under various loading condition. Among them addition of functional nanomaterial in epoxy matrix have consistently exhibited significant range of improvement in its tensile and fracture behavior [14–18].

In recent day's nano material incorporated polymer matrix composite (PMC) have got yielded cumulative interest amongst the people working for enhancements owing to its better physiochemical properties that significantly differ from nano-sized particles in the midst of those bulk molecular materials. These nanomaterial reinforced composite materials synergistically combine the properties of both base polymer and filler nanomaterial [19–22]. This novel approach helps in to improvise the basic and as well the functional behavior of thermoplastic and thermosetting polymers. Also, it is under substantiation that a minimal loading of nanomaterial in PMC show confirmations for considerable improvement in its desired properties. Various types of inorganic nanomaterial viz. Al_2O_3, SiO_2, TiO_2, ZrO_2, graphene etc., have been tried up by different personalities just to improve the mechanical

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behavior of PMC [23–28]. Derivative of Graphene namely, Graphene Oxide (GO), Graphene Nanoplatelets (GNPs), reduced Graphene Oxide (r-GO), graphene nanosheets etc., poses outstanding properties majorly due its higher surface area [29–38]. In precedent years, Graphene based materials comprise for introductions into an extensive choice of polymer matrix; the list not only includes epoxy, polystyrene, polypropylene but also nylon for diverse yet extensive applications. These layer based graphene fillers has established a capability of mechanical toughness improvement due to its unique properties. TiO_2 based nanomaterial has been comprehensively used in industries such as electrode materials, catalysts, solar cells etc. Nanomaterial reinforced thermoplastic composite showcase better basic and functional properties [39,40]; on the other hand, the same using thermosetting based polymers have not been deliberated in and out, that too particularly using TiO_2 though there are few researches in which graphene and TiO_2 based materials are used as filler materials just to improve the functional behavior of polymers.

Vacuum assisted resin transfer molding was adopted by M.Rafiee and co-workers [41] to develop carbon nanomaterial incorporated epoxy/glass fiber PMC. Derivatives of Carbon material such as Multi-walled Carbon Nanotube (MWCNT), GO, GNPs, r-GO are used by them as filler material to improve the thermal behavior of epoxy. Results convey that addition of GNP up to 1% increase the thermal conductivity of base matrix up to 12.6%; and incorporation of GO up to 2% improve its thermal behaviour up to 8.2%. Further, the underwent research concludes that uniform dispersion of nanomaterials enhance its thermal behavior. Zhenyu Wang et al. [42]utilized GNPs to improve the adhesive application of epoxy resin. GNPs incorporated epoxy composite was developed by them and further studied their thick adherence and shear behavior too. Obtained results depicts that addition of GNP in epoxy matrix up to 0.75 wt% exhibited enhancements in shear strength of 102% while compared to that of neat epoxy.

Jaemin Cha et al. and co-workers [43] made comparative studies on influence of melamine functionalized MWCNT and graphene on mechanical behavior of epoxy. Results reveal that addition of these carbon materials showcase considerable improvement in fracture toughness and as well the tensile strength. Further, the research team also observed that addition of functionalized MWCNT improve the fracture toughness up to 95% and 124% while utilizing the functionalized GNPs. Abdel Rahman Hussein et al. [44] fabricated GO reinforced PMC and investigated further for their influence on cryogenic based treatments over and above the fracture behavior of developed composite. End results perceived that addition of GO initiate better improvement in fracture strain and toughness of the base material. Ultrasonic dual mixing process was adopted by Kaushal Kumar et al. [45] to develop TiO_2 reinforced epoxy composite and thereby examine all of the mechanical plus thermal based properties of the newer material. Accomplished outcomes expose that adding up of TiO_2 beyond doubts improvise the thermal and tensile behavior of base matrix material and also the team further observed that addition of TiO_2 increase the storage modules of epoxy matrix up to 29%.

V.G Nguyen et al. [46] and coworkers developed TiO_2 reinforced PMC and investigated for its influence over that of TiO_2 additions, with respect to property enhancement of base polymer matrix. Answers attained so reveals out that addition of TiO_2 accelerate the basic and functional behavior of base material very well. Based on a thorough and clear cut literature survey, it can well be stated that addition of GO and TiO_2 in PMC helps in improving all of the known basic and as well certain functional behavior of matrix material. Conversely, combination effect of TiO_2 and GO was minimal in the field of polymer matrix composite; hence an attempt have been made to develop GO and TiO_2 reinforced PMC that too with varying wt. percentages of GO viz. 0.5, 1.0 & 1.5 wt% against a constant wt.% of TiO_2.

2. Materials and method

Epoxy resin diglycidyl ether of bisphenol A grade LY 556 along with HY951 grade ($C_6H_{18}N_4$) hardener possessing 2–3 h gel time in general was used as the matrix. Utilization of the said material is recommended as because of lower viscosity range, fiber impregnations in addition to appreciable dimensional stability. To ensure progress towards mechanical behaviors of the matrix (specifically epoxy here) GO and TiO_2 were considered as the filler materials. Synthesis for GO and TiO_2 is followed procedurally as already reported in our previous journal descriptions [47,48]. Based on available literature it is highly notable that addition of TiO_2 up to 2 wt% improvise the functional properties of base material; any further increment in TiO_2 might exhibit a decremented trend over their inherent properties. Possessing these considerations, in this research also wt.% of TiO_2 was minded to be unchanging (2 wt%) [49]. GO was varied up to 1.5 wt% only; even though possible beyond this limit any further increment in GO usually showcased lower strength due to the agglomeration tendency of GO. The novel hybrid composite is then fabricated by adopting compression-molding route. Herein the calculated volumes of filler particles are uniformly dispersed into ethanol by the way of ultrasonication process maintained atleast for an hour or so. After that, dispersed GO/TiO_2 particles were added along with epoxy resin at room temperature and then this mixture was treated by means of mechanical stirrer for 1 h at 1400 rpm in order to attain uniform dispersion and as well for the evaporation of acetone to take place. The mixture was further degassed in a vacuum bucket for 45 min approximately. Finally, the attained composite mixture was poured into a flat die of 300 × 300 × 3 mm dimension and then allowed to cure for 24 h at room temperature itself. The compositions of the developed PMC are as depicted in Table 1.

3. Material characterization

X-Ray Diffractometer (XRD - BRUKER D8 model) was used to confirm the presence of filler materials. The developed composite were scanned at 2 theta range of 10° to 80° with K-Alpha of wavelength of 1.5406 Å. Instron made computerized universal testing machine was used to understand the behaviors based on tensile in case of the novel composite developed. Under ASTM D 638 standard, these samples were pieced through abrasive water jet machine in the form of a dumbbell (a usual shape supposed to be with a cross head) by maintaining a speed of 2 mm/min and a gauge length of 5 mm as parameters for general machining. Flexural strength and flexural modulus of the composite are measured using ASTM D 790 standard, with specimen dimension of 125 × 13 × 3 mm. ASTM D 256 standard, was adapted for understanding the impact strength behavior, followed on using the Charpy impact testing machine with sample dimension of 65 × 13 × 3 mm. Each experiment was repeated for atleast three times and then the average value of the measurements has been taken for the sake of exactitude of results. Fracture surface of specimens are checked up by utilizing scanning electron microscopy (SEM) being advanced in technology, this machine was coupled with a schema of energy dispersive X-ray analysis too (SEM-EDAX) (JEOL JSM6610LV, Japan); the machine was usually being operated at 20 kV. Hitachi STA7300 model Thermal Gravimetric Analyzer was utilized to analyze the thermal stability and thermal degradation of the developed composite samples. Herein, weight of the samples was taken as 2.48 mg for calculating the weight loss percentage with respect to temperature. Thermogravimetric analysis and as well the

Table 1
Composition of developed composite.

Designation of composites	Composition
PLATE 1	Epoxy+2 wt % TiO_2+0.5 wt % GO
PLATE 2	Epoxy+2 wt % TiO_2+1 wt% % GO
PLATE 3	Epoxy+2 wt % TiO_2+1.5 wt % GO

Derivative thermogravimetric analysis was carried out for a temperature in general ranging from 28° to 500 °C that too in a gas atmosphere (nitrogen) environment along with a heating rate (as per the suitability of around 20 °C/min) using an alumina pan.

4. Results and discussion

4.1. XRD analysis of composite sample

XRD of the developed polymer composite (plate 1) is as depicted in Fig. 1. Herein the peaks near 25.4°, 38.1°, 48.2° are believed to be the respective peaks of Titanium di oxide with JCPDS card no: 98-010-6862 with crystallographic planes of 011,004,020. Narrow peak near 55.3° was anatase phase peaks of Titanium di oxide with 121 planes. Remaining peaks are the peaks related to epoxy and GO.

4.2. Tensile strength of developed composite

Fig. 2 depicts the variation of ultimate tensile strength with respect to GO addition. It can be seen that increase in concentration of GO exhibits incremental strength. Higher tensile property of 21 N/mm^3 was attained for 1.5% of GO. The ultimate tensile strength of developed composites with GO has significant change for tensile properties. Hardly, strong enough interfacial bond involving the filler materials and as well the matrix in and around is very fundamental to determine the inherent property of PMCs. However, interfacial bonding between nano fillers viz. GO, TiO_2 and polymer resin is better and hence transfer of load from that of the matrix towards the filler materials was always higher. Yet another reason might get associated with that of the structure of TiO_2 and GO; eventually that will avoid the chance of slipping effect from occurring. Further, presence of these nano-fillers in epoxy exhibits substantial curvatures and further upon loading, Nano fillers needs to be extended and thereby rotates down in the direction of tensile in order to exploit their enhancements over the tensile modulus. This improvement in tensile strength is endorsed for attaining better adhesion strength between the filler material and epoxy matrix thus helping in constructing reduced void content in fabricated composites [33,42,45].

4.3. Flexural properties of developed composite

Variation of ultimate flexural strength with respect to GO addition was displayed in Fig. 3. As known, betterment in case of properties relating to flexural usually combines along with nano fillers; herein, all such fabricated composites were completely endorsed to interphase between that of the fillers and matrix, which upholds for interfacial adhesion between GO,TiO_2 and as well the epoxy matrix by means of chemical and mechanical bonds. In general, increase in flexural modulus results in decrements of the subjected materials ductility and further a substantial deformation will occur before that of the material failure commences. The flexural strength of composites is more than the tensile strength as since in case of flexure, the most durable composite itself tends to break. However, improvement in strength was notified for GO while loading at 1 wt % and as well any further addition of GO exhibited only decrements in value.

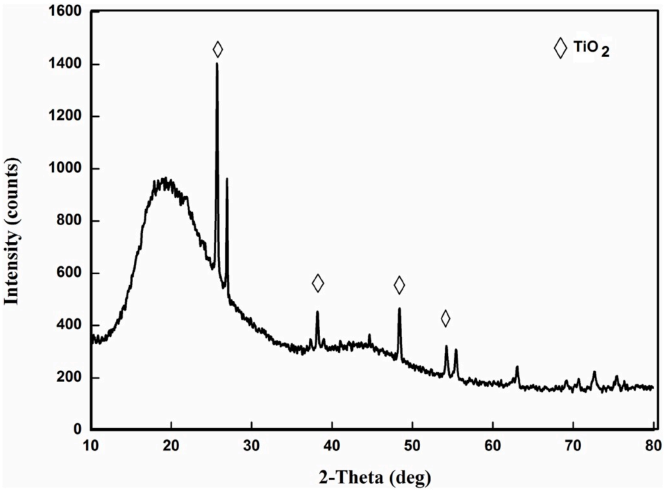

Fig. 1. XRD of developed composite.

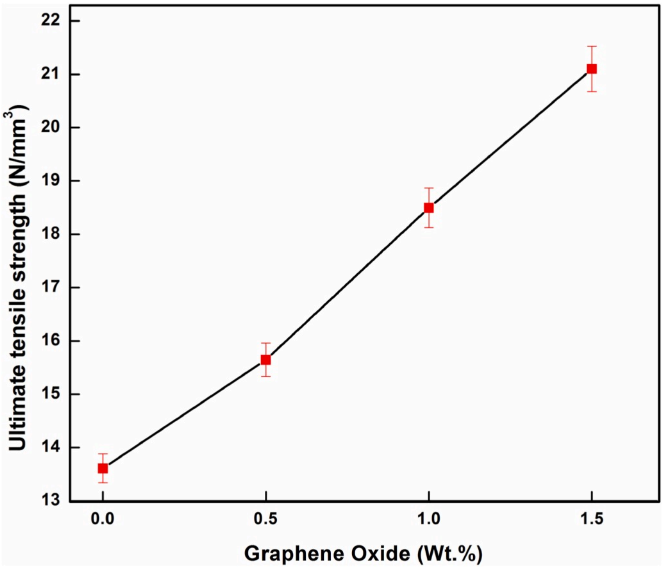

Fig. 2. Tensile strength of developed composite.

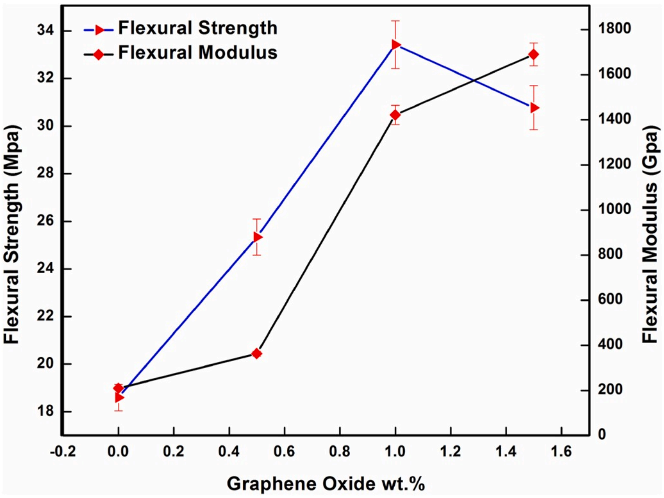

Fig. 3. Flexural strength of developed composite.

In support of flexural modulus along with strength, sort of epoxy interactions by means of nano level filler materials disclose uppermost level of GO (here as 1 wt %) thus emerging as a strengthened material. Yet again, any further raise in nano level filler materials might eventually cause for decrease in flexural strength, that propagate to structure out a material with enough ductility. Observance from that of the attained failure manners, specifies that delaminations were out warded over and above the surface of newer composite (Fig. 6c) samples [50–52].

Higher volume of interfacial disconnections were observed over the composite (Fig. 5), and further certain type of mechanisms related to

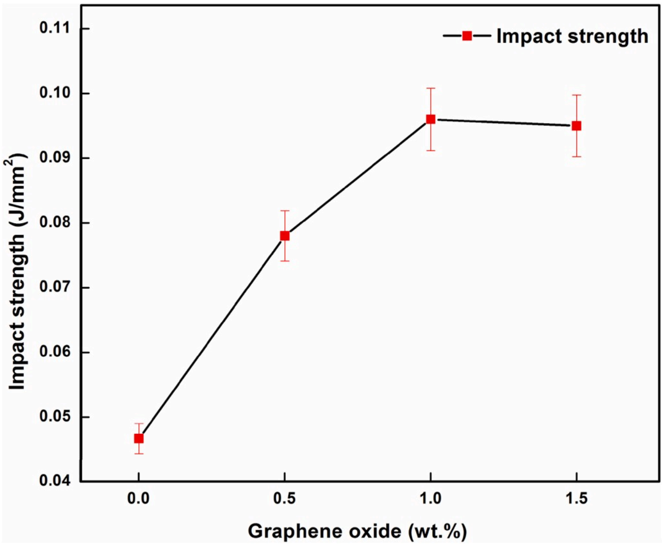

Fig. 4. Impact strength of developed composite.

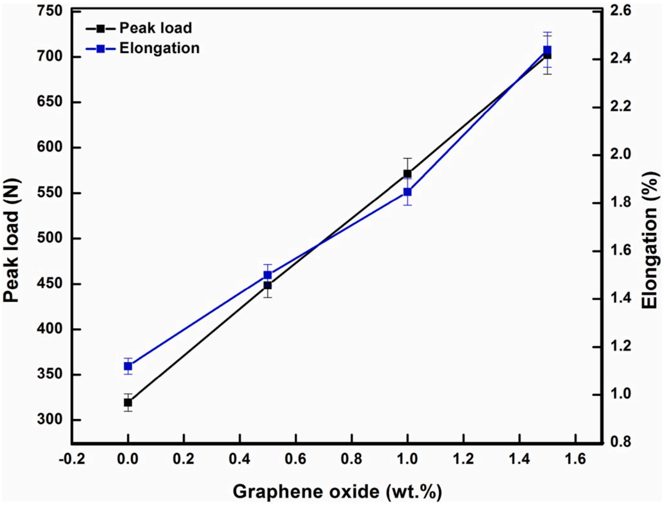

Fig. 5. Elongation and peak of developed composite.

failure that are indicative over the composite sample surface have more no:of pull outs revealing the truthiness of weak bonding (here interfacial in nature) between the matrix and nano fillers. While these clauses of weakness have got improvisation at GO loading of about 1 wt %, any further raise of GO substance with that of epoxy will definitely result only in attaining retroactive effects thus lowering the interfacial nature of bonding that in turn increases the void content and thereby cause agglomeration to take place [42].

4.4. Impact strength of developed composite

Impact resistance of materials might be exemplified as taken up energy along with impact strength by means of energy that have got lost while undergoing CHARPY IMPACT TEST module for understanding the fracture behavior of subjected samples. It can be observed from Fig. 4, that impact strength has got increased up to 0.098 J/mm^2 at 1 wt % GO content. This might be due to the incorporation of nano fillers into the epoxy matrix that assist in attaining a better chemical bond amid the matrix along nano based fillers. Herein, the nano fillers incorporated epoxy matrix allows ample space for molecules in order to distort them to absorb energy during impact testing. Another reason owes to the better rate of adhesion and as well larger surface area of GO particles; these reveals out for better resistance against bending thus exhibiting superior ability to absorb energy near the impact regions. For polymer based composites, matrix cracking is supposed to be the main damage that might occur during impact analysis. From Fig. 4, it will be capable of knowing about impact strength of the newer composites specifying that there is increase along with addition of GO filler up to 1 wt %; any further addition will tend to deliver a gradual decremental trend only.

Impact strength of 0.5 wt % GO showcase a lower impact strength range when matched up to some other samples developed so. It is made known that raise of impact strength can be recognized as an end effect of perfect bonding maintained between the GO material with that of matrix by way of TiO_2 particles; this will on condition leads to more energy absorption capability over and above having much stronger plus stiffer properties when judged against other such sample pieces. In consequence, it is believed for the existence of a satisfactory transfer of loads among GO - TiO_2 – epoxy and thus make available the developed composites to endure with high rate of impact based loadings. Also, strong interfacial strength is generally dependable for the quantity of TiO_2 pull-outs reinforced by huge sum of matrix (polymeric base). Even if comparable, impact strength performance in addition to damage mechanisms be experiential at 1 wt % and 1.5 wt %; TiO_2 based pull-outs at 0.5 wt % were commonly greater than those of the GO loading that happens at 1 wt %. These artifacts imply that any further inclusion of GO once above 1 wt % might facilitate to have lessening of interfacial bonding among the matrix material with GO filler; furtermore, this observable fact show the ways to cut short the impact strength along with delamination too [6,7].

4.5. Peak load and elongation

Fig. 5 displays the variation of elongation and peak load constructed with respect to GO addition. These values are used to analyze the brittleness and strength of the fabricated composite for various weight percentages as decided. Peak load and elongation of composite get increased whenever adding GO fillers in epoxy matrix takes place; this might owe to the fact of interfacial adhesion supported by load transfer capability of GO as a filler.

4.6. Fracture surface analysis of developed composite

Fracture surface morphology of the developed samples are showcased here as Fig. 6a-d. Fracture surface of the neat epoxy (Fig. 6a) is unremarkable that too with a representative brittle fracture process; as well the same is also very much smooth thus disclosing its true nature i. e., the weaker resistance characteristic towards crack initiation and further propagation. From Fig. 6b, the micro voids identified might be due to the debonding effect of nano filler from the matrix that results in fall outs and thus leading to plastic deformation. Plastic void growths have been considered to be one of the major toughening mechanisms for nano filler/epoxy composites. In this, the occurrence of void might be due to the effect of increasing order of toughness transformation along with micro cracks generation. Whatsoever, lower interfaces amid the filler and matrix usually consequence for the pull out effect of nano filler; this is due to increment in stress that in turn heads in to reduce interfacial bonding within the composite. Rough surface fracture morphology with lots of ridges designates the occurrence of ductile fracture mechanism (Fig. 6c). Pull out over the fracture surface are observed to be minimal and this fact owe to the presence of sufficient quantity of nano filler. It proposes that existent of a better filler-matrix interfacial bonding promote phenomenon's like dispersion to happen as an end result of a chemically functional group prevailing over the surface of GO. It is better said that a strong interface as well improvised dispersion will experience a significant influence upon the major mechanical properties of the subjected composite materials. This further suggests that the functionalized GO in matrix material discourage the

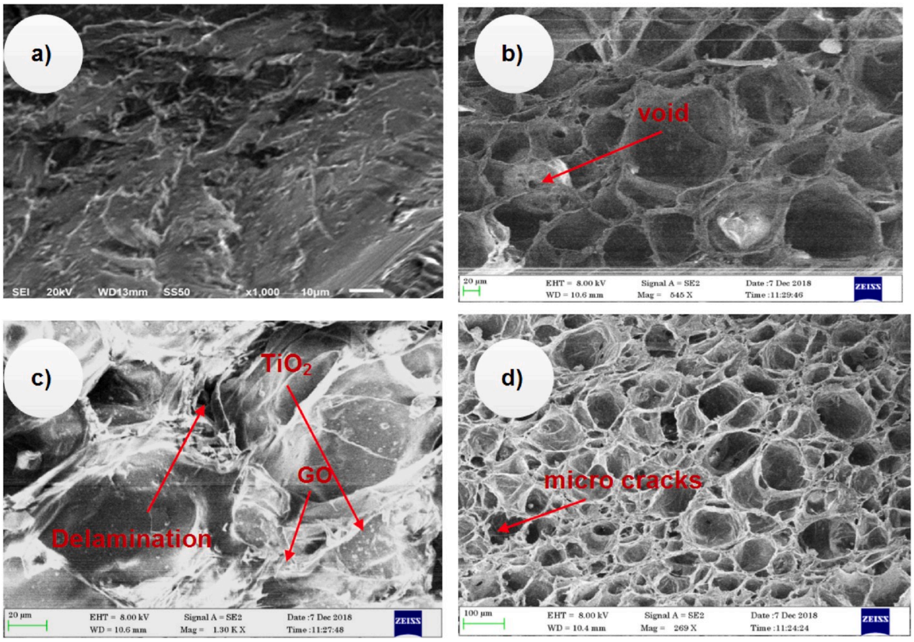

Fig. 6. Fracture surface of a) Neat Epoxy b) 0.5 wt% nano filler c) 1 wt% nano filler d) 1.5 wt% nano filler.

fractures, that bring up to dissipate fracture energy. Relatively smooth fracture surface in the track of the force leading to fracturing are evident through Fig. 6d. But however, the same is not uniform; such observances mean that in all these regions, the surface is supposed to be mirror-like and will put forward a irregular distribution and as well dispersion of nanofiller within the epoxy matrix. On the other hand, some gaps between GO and matrix can be also be found on the surface that designate weaker interface between filler and matrix. Further debonding/delamination along with crack bridging of GO were considered to be the critical elements for the toughening outcomes in composites [33,34].

Thermo-gravimetric analysis of developed composite:

Thermal analysis of composite with 1.5 wt% GO are as depicted in Fig. 7a-b. Since GO exhibit better thermal stability, samples with maximum addition of GO was considered for thermal analysis; the same were usually done in order to understand the influence of GO over matrix material. Fig. 7a displays the mass loss of samples with respect to temperature. Herein, the thermal degradation of sample takes place in three stages based on incremental range of temperature. In initial stage the degradation occurs from 30 °C to 370 °C with 23.3% as weight loss percentage. Second stage of thermal degradation started at 373 °C; it further got rapidly decreased with 63.3% of mass loss. Final stage of degradation ended at 470 °C that too with 91.4% of mass loss. Thermal degradation of composite with respect to residual mass was calculated from Fig. 7b and is as depicted in Table 2.

Thermal stability of the newer composite at T_5 was up to 142.01 °C; when T75 was put up in limelight weight loss happens at 410.09 °C. Herein, derivative thermogravimetric value is 994.68 μg/min and maximum DTG temperature is supposed to be 379.32 °C for that of the newer composite developed comprising with a residual yield of 8.75%.

5. Conclusion

This study reported the synergistic improvement of epoxy derived polymer composites developed through compression molding route reinforced with GO plus TiO_2 particles besides understanding their mechanical behavior. Observed results of the so developed novel hybrid composite may be stated as:

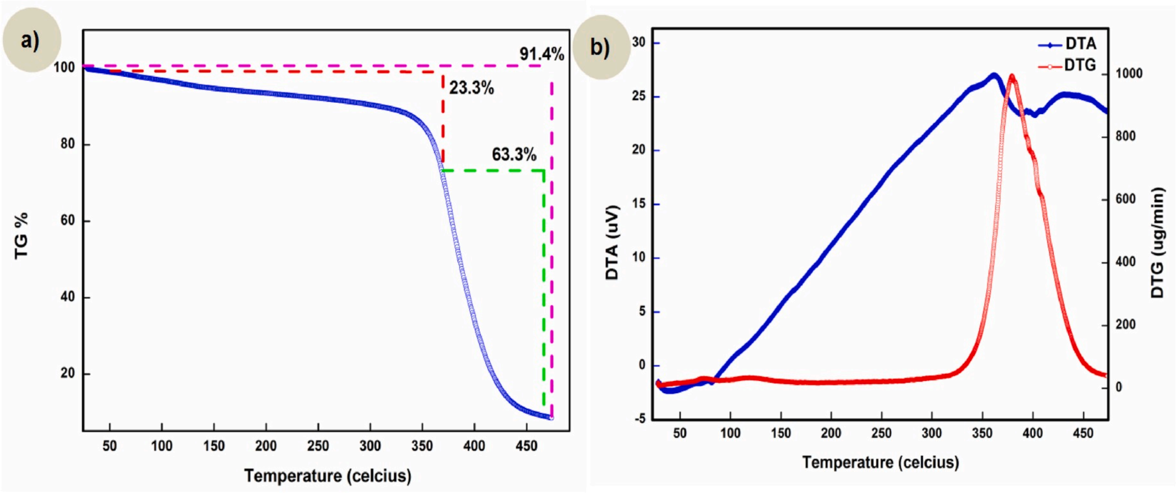

Fig. 7. (a–b) Thermal Analysis plot (TG %, DTA,DTG) for composite with 1.5 wt% GO.

Table 2
Thermal degradation of developed composites with 1.5 wt% of GO.

Composition	T5	T25	T50	T75	DTG(ug/min)	TDTG max °C	Char yield% at 470 °C
Plate 3	142.01	366.98	385.67	410.09	994.68	379.32	8.75

- Tensile strength of the epoxy matrix increases from 13.6 N/mm^2 to 21.5 N/mm^2.
- For the utilized epoxy matrix, Flexural strength raise from 18.6 Mpa to 33.4 Mpa; as well flexural modulus changes from 210 Gpa to 1700 Gpa respectively.
- Peak load of the epoxy matrix increases from 350 N to 700 N with 2.5% of elongation percentage.
- Impact strength of the epoxy matrix enlarges from a known value of 0.045 J/mm^2 to 0.096 J/mm^2.
- Fracture mechanism of developed composite depicts that crack bridging in case of GO may possibly be one of the vital element for toughening effect to happen in newer composites.
- These improvised and strengthened composite can very well be used in for many structural and house roofing applications.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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