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Studies on wood carbons / unsaturated polyester composites

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ABSTRACT

General purpose unsaturated polyester resin (GPR) specimens containing carbon (Teak wood, Ground nut, Neem wood and Rose wood) prepared from environmental waste were fabricated using casting technique. Fabricated composite materials were subjected to various studies viz., Thermal (TGA/DSC), mechanical (hardness), morphology and X-ray diffraction studies. SEM images were carried out to identify toughening. The inadequacy of the bond between filler and the matrix show minor particle agglomerations in specimens containing higher weight percentage (40%) were believed to have responsible for a consistent decrease in property especially the mechanical properties like hardness. Results of the studies implies that neem based carbon found to have superior in properties than that of rest of the carbon based composite in overall properties.

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KEYWORDS

Wood carbons;
Polyester resins;
Epoxy composites.

INTRODUCTION

A composite material is made by combining two or more materials to give a unique combination of properties. Composites based on a polymer matrix have become more common and are widely used in many industries due to the advantageous properties offered by the polymers. Filling polymers with mineral dispersion has long been a practice in the plastic industry as a way to reduce overall production costs and enhance certain properties^[1]. Advancements in material performance depend on the ability to synthesize new materials that exhibit enhanced properties such as strength, fracture toughness, impact resistance, durability etc., Compos-

ites are ideal materials to meet this challenge, as it has been shown that they have the potential to deliver the aforementioned properties oftentimes with minimal increase in weight: a luxury not always realized with conventional composites or metals. It should be pointed out, however, that the degree of enhancement of a particular property is highly dependent on the matrix/filler type system used, the extent of filler adhesion to the matrix and the level of dispersion of the filler throughout the matrix.

Polymer resins have been used extensively as matrix materials for many high performance components in the aerospace, automobiles and electronics industry because of their mechanical, electrical and chemical

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properties. Highly cross-linked thermosetting polymeric materials, such as the unsaturated polyester resin used as the matrix material in this study, are extremely brittle owing to their covalently bonded network structure, and thus are poor inhibitors of crack initiation and propagation. Nonetheless, researchers have been able to improve their toughness thus obtained is often accompanied by a concomitant decrease in modulus and strength^[1-4].

Generally, most mineral fillers used in thermoset and thermoplastic composites are ground into fine particles with relatively low aspect ratios. The low aspect ratios and relatively low price of the fillers are very attractive in a plastics market that grows more and more competitive^[5]. Almost any powdered material can be used as filler, the common ones being obtained from natural deposits. Of the several hundred fillers used, those that find widespread use are various grades of calcium carbonate, quartz, mica^[6], silica flour, talc^[7], and various clays^[8]. The utilization of waste fly ash material as an additive component in polymer composites has received increased attention recently, particularly for price-driven/high-volume applications^[9]. The development involves, the utilization of carbon prepared from environment finds an alternative to fly-ash and other fillers has been brought about because the incorporation of carbon from renewable offers several advantages; because it is the best way to dispose the waste carbon. It is a fine and powdery material. It has been used as a filler^[10] for the production of light-weight and high-strength composites^[11] these fillers have been shown to increase the stiffness of the composites, but the strength, however, suffers a setback^[12]. Srivastava and Shembekar^[13] evaluated tensile and flexural properties of fly-ash-filled epoxy resin, and they reported that the loading of FA in epoxy-resin causes a decrease in the tensile and flexural properties of the composites. A wide variety of fillers have been incorporated in pure polypropylene (PP) to impart flow and mechanical properties and to reduce costs^[14]. Chand and Gautham^[15] developed composites of FA and glass fiber with polyester resin and reported their abrasive behavior and wear loss. Coutinho et.al^[16] prepared a composite of wood fiber and PP and found a decrease in mechanical properties. Many studies have been published concerning the processing conditions and properties of thermoplastics with

wood fiber^[17-19], glass fiber^[20,21], mica^[22], and calcium carbonate^[23]. Unfortunately, the better stiffness obtained through filling is often accompanied by drawbacks such as lower processability and lower toughness.

Enormous amount of research work focuses on various fillers on unsaturated polyester composite were found to have deteriorated in overall properties of the composites. The reports on the utilization of carbon prepared from environmental waste found along with unsaturated polyester found to have very less. Hence, the goal of the present investigation involves the fabrication and comparison of four different environmental waste carbon as fillers prepared from environment waste /unsaturated polyester composites. The spectral, mechanical, thermal and morphological characterizations of these composites have also been discussed.

EXPERIMENTAL

Materials

General purpose unsaturated polyester resin (GPR) is composed of maleic anhydride, iso-phthalic anhydride, aliphatic diol, styrene monomer, 1% solution of methyl ethyl ketone peroxide (catalyst), 1% solution of cobalt-naphthenate (accelerator), and surface-modified calcium carbonate filler (120–150 m, bulk density 50.7289 g/cc) were obtained from Sakthi Fiber Glass Ltd., (Chennai, India). The carbon prepared from various renewable sources like Neem wood, Teak wood, Rose wood, Ground nut wood in a pre-dried powdered form were used as filler in this investigation.

Fabrication of composite sheets

Carbon particle prepared from environmental waste were powdered and added in varying weight percentage viz., 10, 20, 30 and 40 % respectively to 90. 80, 70 and 60 weight percentage of general purpose unsaturated polyester resin, and mechanically mixed in a glass beaker for approximately 10 minutes. The mixture was placed in a vacuum chamber at 28 Torr for 5 minutes to remove trapped air bubbles generated during mechanical mixing process. After the deaeration process, the catalyst methyl ethyl ketone peroxide (MEKP) and the accelerator Cobalt naphthenate (CONAP) were added separately to the mixture and mixed thoroughly at 1 ml of 1% solution y weight of the

polyester, respectively, to initiate and accelerate the free radical polymerization process. The mixture was then deaerated at 28 Torr to remove trapped air bubbles generated during the mixing process. The second de-aeration process was necessary as free radical polymerization commences almost immediately after the catalyst and the accelerator is added^[24]. The final mixture was poured into the mold and rotated at 3 rpm for at least 6 hours to avoid the settlement of the carbon particle at the bottom. The resin mixture was allowed to cure at room temperature for 24 hours. At least six specimens of each type were made and subjected to testing to obtain the average value for hardness properties and to avoid possible errors obtained because of non - uniform distribution of the fillers. The hardness (ASTM D2240) of the composite was studied with the durometer / shore "D" hardness tester (Blue Star Engineer's pvt. Ltd., Bombay, India).

Characterization of carbon

The carbon was characterized for the following properties: Moisture content, loss on ignition and pH. The notations of various environmental waste carbon and there composites are presented in TABLE 1. The results are given in TABLE 2 The particle size of the wood and their concern environmental waste carbon

TABLE 1 : Characterization of carbon

Parameter	Concentration (Average)			
	C1	C2	C3	C4
Moisture content (%)	6.4587	6.9508	5.8537	7.2376
pH	7.4000	7.200	7.6000	7.1000
Bulk density (g/cc)	0.8487	0.7437	0.8678	0.7982
Loss on ignition (%)	0.9566	0.8506	0.7543	0.8592
Sulfate content (%)	0.2023	0.2961	0.3333	0.1307
Chloride content (%)	0.3090	0.3292	0.4210	0.3828

TABLE 2 : Particle Size

NAME OF THE WOOD	SIZE OF THE PARTICLE (%) (BSS 40 - 425 μ m)	SIZE OF THE PARTICLE % (BSS 60 - 250 μ m)
Teak Wood	88.0859	70.5657
Groundnut	35.0112	57.3495
Neem Wood	64.5647	63.3031
Rose Wood	53.2606	69.6921

were determined by sieving through a suitable sieve (standard test sieve BSS 40 to 425 μ m and 60 to 250 μ m). The carbon with particle sizes in the range of 60-250 μ m was used in this study.

Thermal studies

In order to evaluate the thermal stability understand the phase transformation in the prepared samples. The TGA studies were performed in the range of ambient to 870°C and the results are shown in the figure. All TGA analyses were conducted using a TA Instruments TGA Q500 V20.10 Build 36 thermal analyser, using constant heating rate (20°C/min).

Morphological studies

SEM studies

Fractured surface was subjected to gold coating using sputtering technique and analyzed by SEM. The analysis is carried out VEGA3, TESCON, GERMANY.

X-ray diffraction

X-ray diffraction patterns were obtained using a Bruker D8 advance diffractometer equipped with a Cu source (wavelength 1.54 nm) operating at 40 kV and 40 mA. Scanning rate was 0.02° s⁻¹ from 2 θ = 2° to 20° for various carbon samples and 2° to 40° for calcium carbonate samples to check for alteration in the characteristic diffraction peaks of the material.

RESULTS AND DISCUSSION

Thermal studies

Thermogravimetric analysis was employed to get information on thermal stability of the prepared composites. It can be observed that all samples have undergone two stage decomposition with the major weight loss occurring between 300 to 588°C. the peak temperature and the percentage of residue of all the samples for the both decomposition are listed TABLE 3 and Figure. 1 - 4, summarizes the TGA data on C1, C2, C3 and C4 GPR composites. The 10% weight loss neat GPR, C1/GPR, C2/GPR, C3/GPR and C4/GPR was observed at 276°C, 230°C, 208°C, 215°C, and 210°C respectively. The 20% weight loss found to have around 300°C for all composites. Similarly 50% weight loss was observing around 400°C. The maximum (70%

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weight loss have been observe C1, C3 and C4 at around 580°C, 600°C whereas C2 was found to have only at 510°C. this might be due to the lesser hardness imparted by the C2.

TABLE 3 : Thermal stability of GPR and modified Carbon/GPR composite systems

Composite	Weight loss (%)						
	10%	20%	30%	40%	50%	60%	70%
GPR	276	374	392	406	464	538	620
C1/GPR	210	300	350	375	400	425	695
C2/GPR	195	255	340	380	400	500	630
C3/GPR	210	300	350	400	412	450	630
C4/GPR	210	305	360	380	400	420	610

Scanning electron microscope (SEM)

In order to study the morphology of C1, C2, C3 and C4 with polyester composites were examined by Scanning Electron Microscope. These samples were

subjected to Ag/Pd coating to render these conductive before examination. Polymer rich phase was shown in the top. Particles can inhibit crack initiation and propagation if dispersion is such that mechanism like crack trapping and pinning are allowed to occur. Crack trapping, in particular, can be promoted by ensuring that there exists a strong bond between the filler and matrix. Although not used in this study, silane has been used for this purpose^[4]. Agglomeration, on the other hand, can behave as crack initiation sites, lowering the fracture toughness of the composites. On increasing the weight percentage of carbon the crack initiation was attributed to an increase, although minor, in agglomeration on increased loading. In other words, as the volume fraction of the particles was increased beyond 40 %, whatever advantages were gained up to this point began to diminish. The fine disposed particles along with agglomerates were viewed in the middle and bottom of

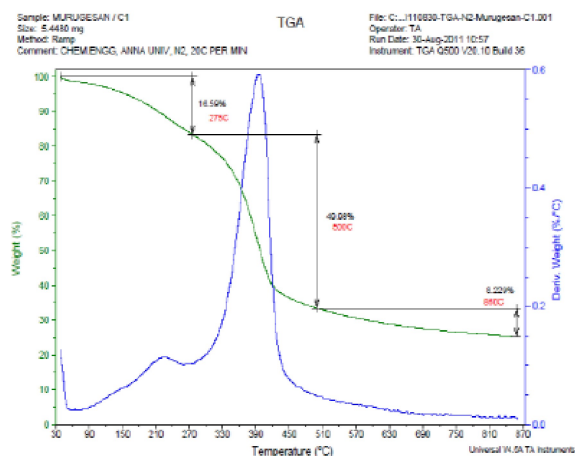


Figure 1

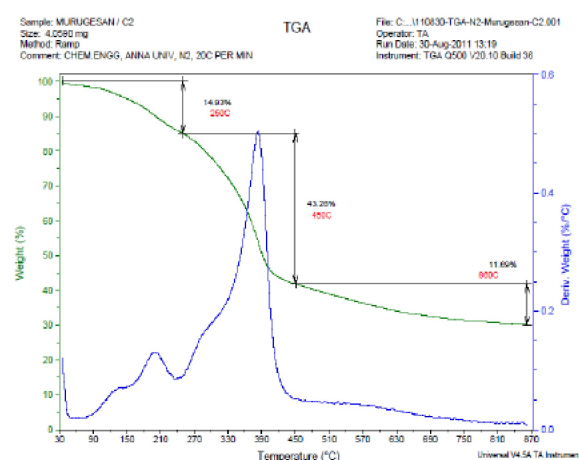


Figure 2

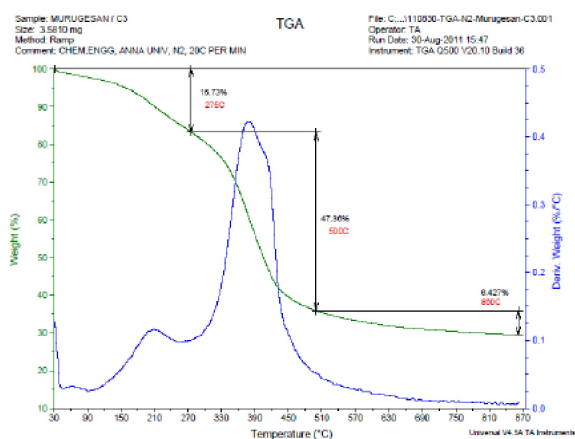


Figure 3

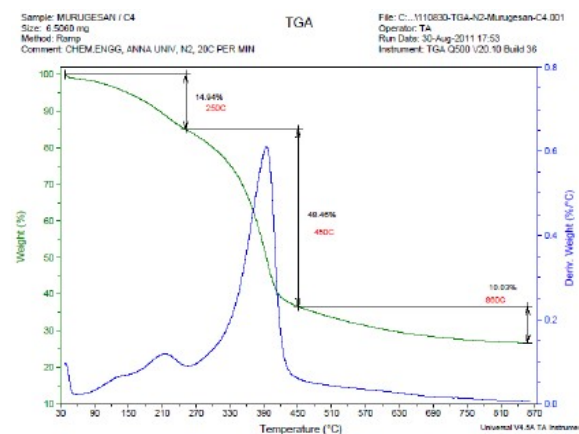


Figure 4

Figure (1–4) : TGA of C1, C2, C3 and C4/GPR Composites

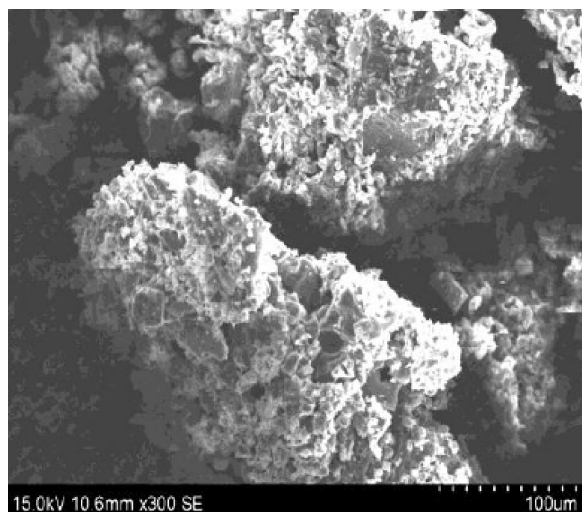


Figure 5

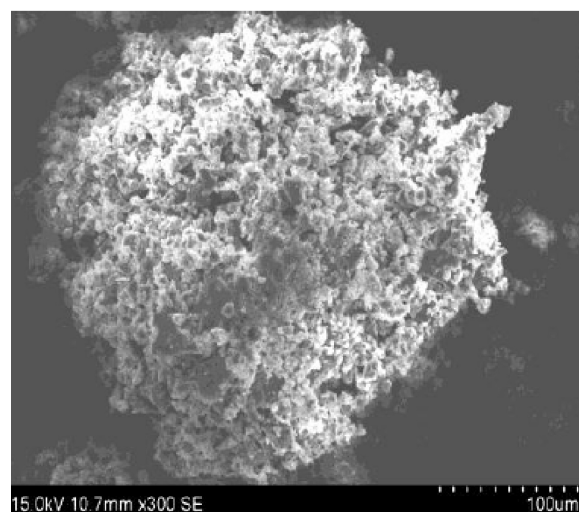


Figure 6

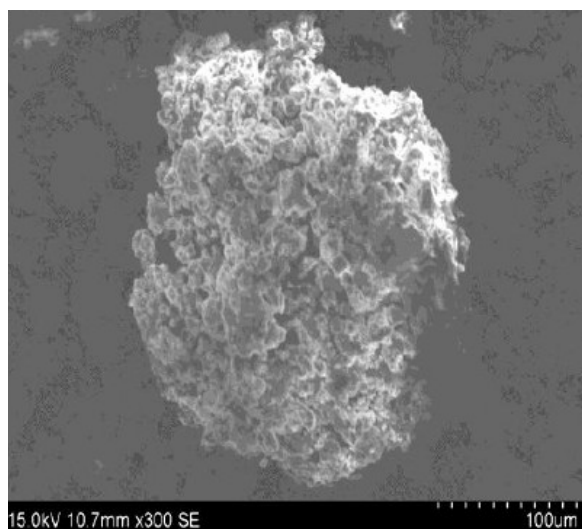


Figure 7

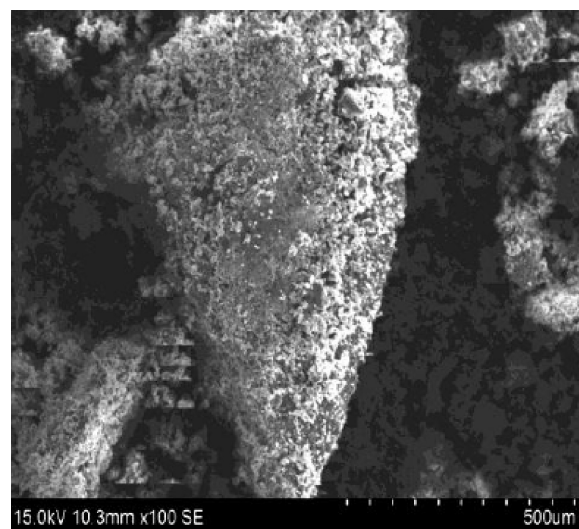


Figure 8

Figure (5–8) : SEM of C1, C2, C3 and C4/GPR Composites

the portion of the C1, C2, C3 and C4 as shown in the Figure 5 – 8. This would substantial phase separation between the polyester and the various carbon prepared from renewable resources and viewed.

Displace the distribution of carbon C1 in the polyester matrix the irregular fracture surface is indicative of proper distribution of C1 matrix, According Lee et al and Gupta et al fractures occurs near the clay particles in the matrix. The Figure C1, C2, C3 and C4 observed to have no affinity with matrix and they do not dispose in the matrix. The possible origins of cracks initiation in a composite material are air bubbles or voids, resin-rich area, foreign matter such as dust particles, particle – matrix poor adhesion (Roulin-Molony, 1987)^[25]. The fractured surface of unfilled resin shows a brittle failure.

Debonding at the interface and subsequent particle pull out are seen in Figure.1-4 for the untreated Carbon/GPR samples which may be due to the lack of proper interfacial adhesion. Another possible mode of failure is agglomeration of the carbon particles are also shown in the figure. Since the carbon particles are randomly oriented, a large number of them are subjected to tensile stresses acting perpendicular to the plane and the crack propagation occurs parallel to the plane.

Effect of carbon on hardness of the composite

The durometer hardness values of the various composites made in this study are given in TABLE 4. From the TABLE 4, it was clear that 15%, 12 %, almost level, and 12 % for C1, C2, C3 and C4 respectively.

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The values indicate that the Carbon-filled composites were harder than the unfilled composites. This observation is in agreement with the fact that the hardness is a measure of resistance to penetration. This resistance to the penetration of GPR increased when filled with the carbon (C1, C2, C3 and C4)

TABLE 4 : Shore D hardness of GPR and Carbon/GPR composites

Hardness test	GPR	C1/GPR	C2/GPR	C3/GPR	C4/GPR
10 %	70.2	76.8	77.6	79.6	74.9
20%	70.5	82.1	84.5	81.5	77.8
30%	70.6	84.0	86.0	75.2	78.6
40%	70.3	81.5	78.8	70.0	79.2

X – ray diffraction

Studies of amorphous materials represent a large and important emerging area of materials science. It is an area which is not amenable to the most of the conventional theoretical techniques of solid - state physics as there is no periodicity to simplify the mathematics. Due to the lack of periodicity, extraction of structural information from amorphous materials becomes very

difficult. In this work we have used XRD technique to analyze the variations of the structural parameters of amorphous carbon due to irradiation. XRD is a very useful and simple technique to understand the structural details of the solid-state substances. Incident X-ray interacts with large volume of the material at a time and an average property of the material can be characterized rather than the local property. This makes XRD a powerful technique for studying the disordered materials which is inherently heterogeneous and where the estimation of average property has got practical significance^[26].

The cured polyester composites containing 40% of either and C1, C2, C3 and C4 was powered and analysed used XRD material Figure (9 – 12) for comparison a diffractogram for physical blend containing environmental waste carbon and polyester in unmodified condition was recorded. The XRD pattern of composites shows amorphous pattern for C1 with $d = 4.268$, $A = 20.792^\circ$, for C2 with $d = 3.37$, $A = 26.417^\circ$ and for C3 with $d = 3.38$, $A = 26.681$ respectively. The presence of peak was not observed in diffractogram of

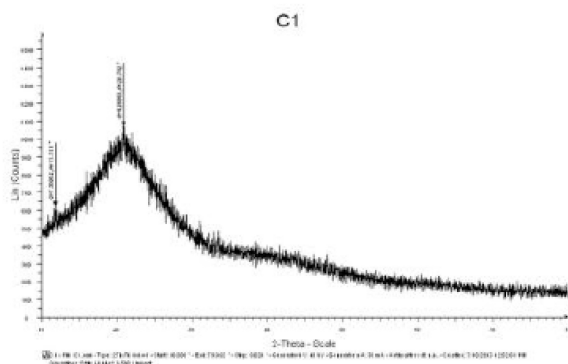


Figure 9

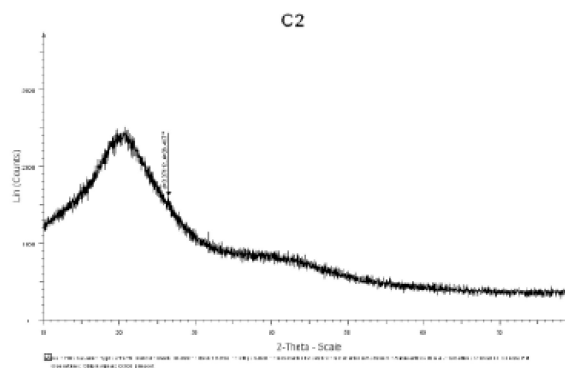


Figure 10

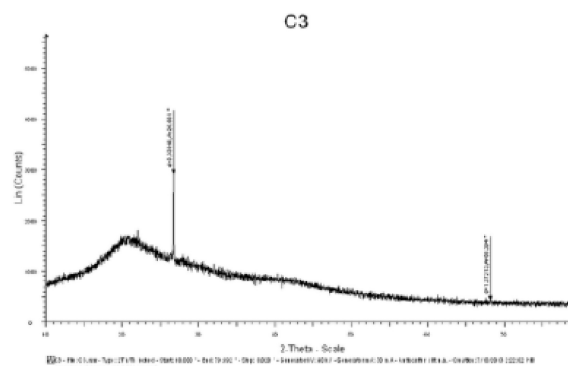


Figure 11

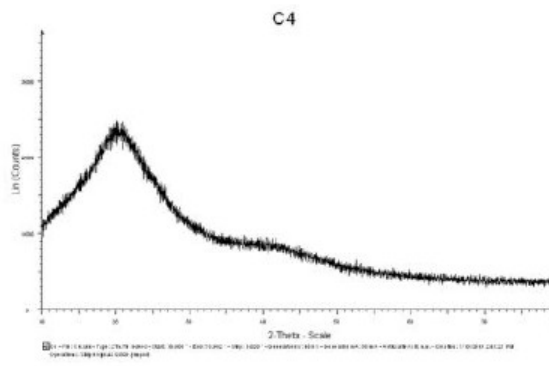


Figure 12

Figure (9–12) : X-ray diffraction of C1, C2, C3 and C4/GPR Composites

composition C4-GPR composite which may indicate delamination of carbon added during polyester fabrication. Similarly to our observation Michal, kedzierski et-al, 2004 was identified for the polyester/MMT nano composites preparation using intercalative copolyaddition reaction^[27]

CONCLUSIONS

In summary, four different carbon prepared from environmental waste with general purpose unsaturated polyester composites were fabricated using casting method. XRD pattern of all composites shows amorphous in nature. Thermal studies reveal that two stage decomposition were observed for all carbon/GPR composites. The maximum thermal stability for teak wood carbon (C1) based GPR composite (695°C) which was found to have 9.3 % increased thermal stability than the untreated composites. Carbon filled GPR found to exhibit harder than the unfilled composites. SEM images resulted on increasing the weight percentage of carbon the crack initiation was attributed to an increase, in agglomeration on increased loading. In other words, as the volume fraction of the particles was increased beyond 40 %, whatever advantages were gained up to this point began to diminish.

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