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Investigation of the Chemical Decomposition of Periwinkle-Shell Reinforced Polyester Composites

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ABSTRACT

This study investigates the chemical decomposition of periwinkle shell reinforced polyester composite to assess its residue and its possibilities of being recycled. 6M and 8M Nitric acid were added to the composite samples, and the decomposition was monitored by weight reduction. Fourier Transform Infrared Spectroscopy (FTIR), X-ray Diffraction (XRD), and Gas Chromatography - Mass Spectrometry (GC-MS) were used to characterize the composite samples and residue. FTIR of the residue showed the appearance and increase of carbonyl and hydroxyl groups, which indicates that bonds were broken, oxidized, and new functional groups were formed. XRD analysis showed a shift in the major diffraction peak from 21.68° to 25.86° 2θ, with crystallinity increasing from 45.36% to 50.72%, confirming structural reordering and formation of new crystalline phases. GC–MS detected aromatic, ester, and aliphatic fragments. This suggests partial depolymerization and oxidation of the resin matrix. The results indicate that nitric acid is a good hydrolysis and oxidative scission agent in degrading the composite. The work sheds light on recycling possibilities for thermoset composites and advances efforts toward sustainable polymer waste management.

1. INTRODUCTION

Polyester resins are among the most widely used thermosetting polymers owing to their favorable balance of mechanical strength, chemical resistance, and processability. They are commonly used for the production of coatings, adhesives, glass fiber–reinforced composites, and structural materials (Khosravi & Musa, 2011). Their excellent resistance to heat and chemicals is attributed to their cross-linked molecular structure, which provides rigidity and dimensional stability. However, this same three-dimensional cross-linking makes it challenging for them to be recycled for reuse because they do not soften or melt upon heating like thermoplastics (Pickering, 2006; Mallick, 2007). Figure 1 shows the chemical structure of unsaturated polyester resin.

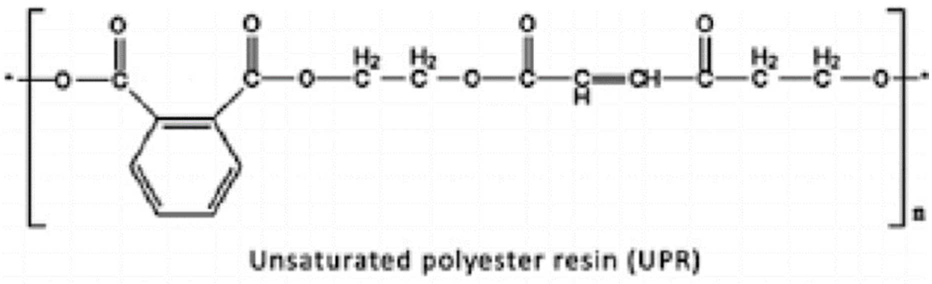

Figure 1: Structure of unsaturated polyester resin (Islam et al., 2019)

The disposal and management of thermoset materials have become a pressing environmental concern. Unlike thermoplastics that can be melted and reprocessed by mechanical means, thermosets cannot be melted and end up in landfills or incinerators, contributing to pollution and the depletion of resources (Hamad et al., 2013). The accumulation of nonbiodegradable polymeric wastes, particularly thermosets used in packaging, automotive, and construction applications, highlights the urgent need for efficient recycling or decomposition strategies.

Some approaches have been explored for recycling thermoset composites. Thermal degradation methods, including pyrolysis and gasification, have been widely studied for polymer matrix composites (Ferdosian et al., 2016). Pyrolysis, which normally occurs at temperatures ranging from 400 to 800°C, causes polymer chains to break down into smaller hydrocarbons, gases, and char residues. However, these processes frequently have significant energy requirements, incomplete breakdown, and the production of toxic byproducts. Methods that can be used to chemically decompose them have attracted increasing attention as an alternative due to their ability to provide controlled degradation under milder conditions and facilitate the recovery of valuable monomers or fillers (Khosravi & Musa, 2011).

Several researchers have reported the successful decomposition of thermoset matrices through chemical means. For instance, Hanaoka et al. (2021) investigated the chemical decomposition of bisphenol F–type epoxy resin using nitric acid and demonstrated effective cleavage of C–N and aromatic structures under moderate temperatures. Similarly, Wang and Shi (2006) studied the thermal decomposition kinetics of flame-retardant epoxy systems, showing that activation energy and decomposition behavior strongly depend on chemical composition. Ferdosian et al. (2016) further analyzed bio-based epoxy composites using thermogravimetric and infrared spectroscopy, confirming that incorporating lignin lowers the activation energy and alters degradation pathways.

Other studies have extended chemical decomposition to different thermoset systems. Zoi Terzopoulou et al. (2017) examined poly(ethylene furanoate) degradation using various catalysts and found that titanium-based catalysts significantly accelerated polymer chain scission during repeated heating cycles. Ou et al. (2018) investigated the decomposition behavior of hydroxyl-terminated polyurethane (HTPE)-based composites and reported multi-stage degradation through depolymerization and oxidation. Collectively, these studies show that both thermal and chemical decomposition can be tailored by controlling reaction media, temperature, and catalysts.

However, relatively few studies have examined the chemical decomposition behavior of unsaturated polyester resins, particularly when reinforced with natural fillers. Unsaturated polyester resins are condensation products of glycols and unsaturated dibasic acids, cured into rigid cross-linked networks using monomers such as styrene (Larranaga & Lizundia, 2019). While they provide high strength and durability, their cross-linked structure resists depolymerization, making traditional recycling impractical. Recent interest has turned toward using natural fillers, such as agricultural and marine wastes, to improve both mechanical and environmental performance of polymer composites (Fakhrul & Isram, 2013; Sajan & Philip Selvaraj, 2021).

One such filler is the periwinkle shell (*Littorina littorea*), a calcium carbonate–rich marine shell widely available in coastal regions. Previous studies, such as those by Adewuyi and Adegoke (2008), have demonstrated that periwinkle shells can serve as lightweight reinforcement in concrete and polymer composites, enhancing stiffness while reducing density. Incorporating periwinkle shell powder in polymer matrices may also influence their chemical reactivity and degradation behavior due to the presence of $CaCO_3$ and other mineral components. Understanding how this reinforcement affects decomposition pathways is crucial for developing environmentally friendly composite materials and potential end-of-life recycling methods.

Among chemical decomposition agents, nitric acid (HNO_3) is used as an effective reagent for breaking down thermoset polymers through combined oxidation and hydrolysis. Nitric acid decomposition operates under relatively mild temperatures (below 100 °C) and ambient pressure, avoiding the high energy input required for pyrolysis or supercritical methods (Hanaoka et al., 2021). Moreover, it does not require special functional groups or catalysts, making it suitable for a wide range of resin types. During decomposition, nitric acid attacks ester linkages, hydroxyl, and aliphatic groups, leading to the formation of smaller oxygenated species and stable crystalline residues.

While nitric acid decomposition has been studied for epoxy and fiber-reinforced systems, systematic analysis of its effect on polyester composites containing natural fillers remains limited. Most available literature focuses on fiber recovery or gas evolution rather than the structural changes in the resin itself (Ou et al., 2018; Zoi Terzopoulou et al., 2017). Therefore, understanding the decomposition chemistry, reaction mechanisms, and resultant phase transformations in periwinkle-reinforced polyester resin is necessary for improving recyclability and designing composites that are more amenable to chemical recycling.

This study examines the chemical and thermal decomposition properties of periwinkle-reinforced polyester resin with the use of nitric acid. It targets losses in the material, alterations in its functional groups monitored by FTIR, changes in crystallinity evaluated by XRD, and characterization of the product by using GC-MS. This research is useful in understanding the mechanism of oxidation and hydrolysis of unsaturated polyester resin reinforced by natural fillers by comparing the composition structure of the compound before and after decomposition.

2. MATERIALS AND METHODS

2.1. Material Collection and Preparation of Samples

The materials used for this research include periwinkle-reinforced polyester resin samples, nitric acid (HNO_3), distilled water, and various laboratory apparatus (beakers, conical flasks, a weighing balance, and a water bath oven). The periwinkle reinforced composite samples were prepared at the Department of Metallurgical Engineering, University of Lagos. Figure 2a shows the prepared samples of periwinkle reinforced polyester composites, which were cured at room temperature. Figure 2b shows the experimental set-up which was used to prepare the solution of nitric acid, while Figure 2c shows the set-up which was used to weigh the samples before and after decomposition.

2.2. Chemical Decomposition of Composite Samples

The process of chemical decomposition was performed with the help of 6M and 8M nitric acid solutions. Composite samples were cut into smaller rectangular shapes, weighed, and left to soak in 6M HNO 3 over a period of 23 days. During these 23 days, there was no visible reduction in weight, so the molarity of the nitric acid was increased from 6M to 8M for a period of 8 days. Weight measurements of the soaked samples were done in before, during, and after decomposition to check the effectiveness of the process at recovering the materials. Figure 3a shows the start of the decomposition of the 6M nitric oxide, while Figure 3b shows the decomposition state of the periwinkle reinforced polyester composite samples after 14 days. Figure 4a shows the brown coloration of the solution caused by the decomposition process. Figure 4b shows the composite samples after 4 days of soaking in 8M Nitric acid, while Figure 4c shows an extract of the decomposed residue after filtration.


(a) (b) (c)

Figure 2: (a) Already prepared samples of periwinkle-reinforced polyester composites, (b) Experimental setup in preparation of the nitric acid solution, and (c) Weighing of the samples before decomposition


(a) (a)

Figure 3: (a) Beginning of extract formation in solution due to decomposition (b) Decomposition of the composite in 6M Nitric acid after 14 days


(a) (b) (c)

Figure 4: (a) Brown coloration of the conical flask due to release of NO2 gas during decomposition (b) Composite samples 4 days after soaking in 8M Nitric Acid (c) Filtered residue after Decomposition

2.3. Characterization Tests

Before and after decomposition, FTIR spectra were recorded at the range of 4000 -650 cm to identify functional groups. The variations in the peak positions and intensities were evaluated in order to define chemical changes in the polymer matrix. XRD analysis was performed to examine crystalline structure and determine the crystallinity index (CI) using Equation (1). GC–MS analysis was used to identify volatile and semi-volatile decomposition products, focusing on aromatic compounds, esters, hydrocarbons, and oxygenated species.

$$\frac{Ac}{Ac + Aa} \tag{1}$$

where A_c and A_a represent crystalline and amorphous areas, respectively.

3. RESULTS AND DISCUSSION

3.1. Weight Loss

There was a reduction in the weight of the composite by approximately 23% while using 6M nitric acid, followed by a rapid decrease of nearly 70% when exposed to an 8M acid concentration. The first slow decline results in surface layer dissolving and moderate hydrolysis of $CaCO_3$ and polyester ester bonds at lower concentrations. The reason for the slow dissolution of the composite by 6M is the weak concentration of the nitric acid.

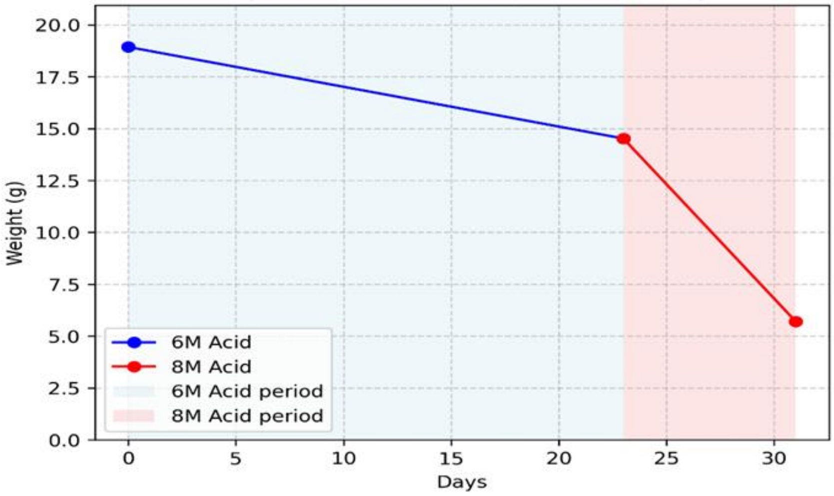

Figure 5: Weight reduction of the composite sample over 31 days

Figure 5 is a graph showing the weight reduction of the composite sample over a period of 31 days. The excess loss in weight that was noticed under 8M acid suggests severe oxidation and chain scission reactions, backed by gas evolution (CO_2) from $CaCO_3$ decomposition as shown in Equation (2).

$$CaCO_3\ (s) + 2HNO_3\ (aq) \rightarrow Ca(NO_3)_2\ (aq) + CO_2\ (g) + H_2O\ (l) \tag{2}$$

This proves the fact that the concentration of the acid has great effect on the kinetics of decomposition whereby the stronger the acidic concentration is, the faster it breaks down the composite matrix.

3.2. FTIR Analysis

Figures 6 and 7 below show the Fourier-transformed infrared spectra of the periwinkle Shell reinforced composite before and after decomposition. Comparison of the FTIR spectra from figures 6 and 7 indicates that the treatment in the nitric acid caused significant chemical changes in the composite. The aliphatic C-H stretch became sharper (2937 to 2946 cm^{-1}), which was probably due to the breaking of the chain and the appearance of smaller fragments. The C=O band, indicating carbonyl-rich end groups, retained the same sharp intensity. This indicates oxidation of the polyester matrix and formation of carbonyl-rich end groups. The fingerprint region (1300-1000 cm^{-1}) of the residue was more detailed and clearer, which indicates the break or cleavage of C-O-C bonds and the formation of new functional groups. The weak unsaturated bands that existed in the composite sample(around 2128cm^{-1} and 2018 cm^{-1}) were also shown to change after treatment, and new absorptions were observed around 2350^{-1} and 2145 cm$^{-1,}$ which suggested either oxidation or the formation of nitrite. C=C and out-of-plane aromatic bands held on, albeit with their intensities mostly reduced (the C=C region becoming smoother), indicating partial perturbation but overall resistance of aromatic areas to react with nitric acid. An overlay of the FTIR spectra for both periwinkle reinforced polyester composite before and after the chemical decomposition is shown in Figure 8.

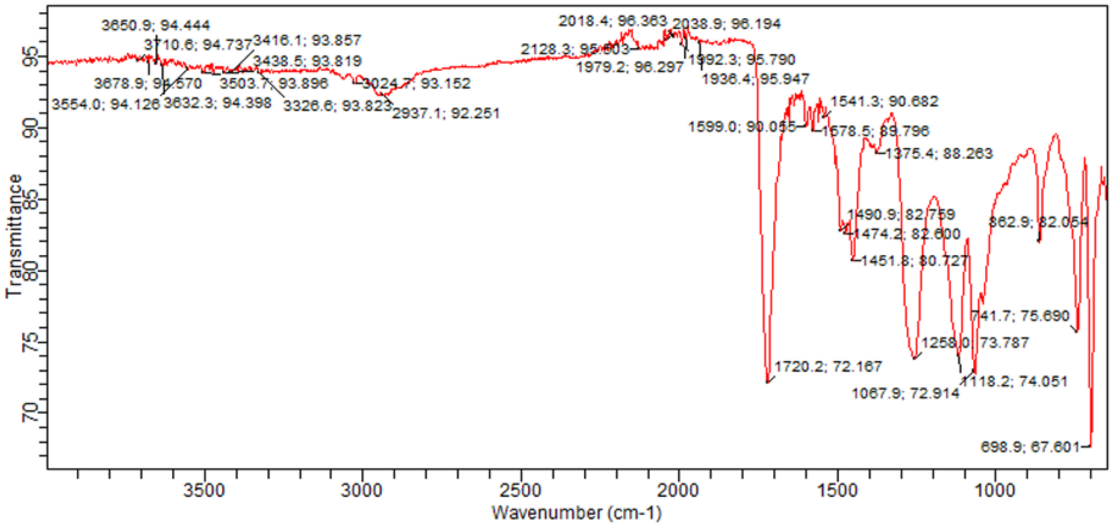

Figure 6: FTIR graph of the periwinkle-shell-reinforced polyester Composite before decomposition

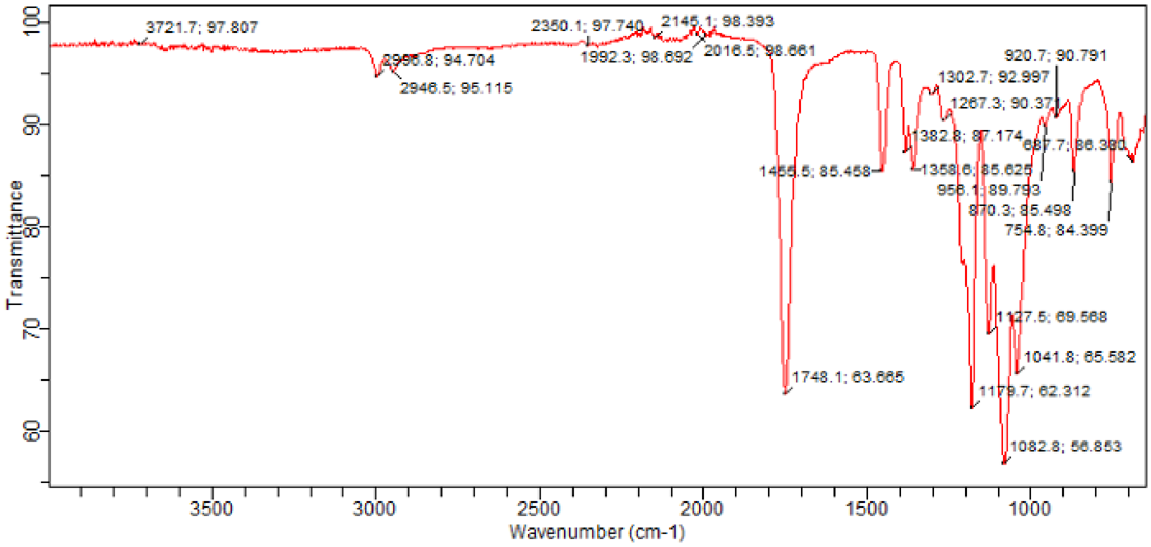

Figure 7: FTIR graph of the periwinkle-shell reinforced polyester composite after decomposition

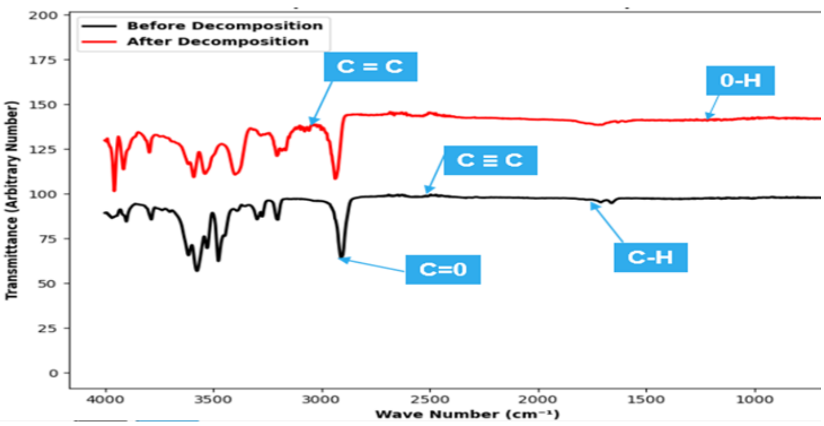

Figure 8: FTIR spectra comparing before and after decomposition

3.3. XRD Analysis

Figures 9 show the XRD results of the undecomposed composite. The results indicated that the composite was a semi – crystalline material with a major peak at 2θ = 21.68° and crystallinity index (CI) of 45.36% . Figure 10 show some of the phases from the XRD. The phases recognized were Fichtelite ($C_{19}H_{34}$), Chaoite (C), Mascagnite ($(NH_4)_2SO_4$), and Silicon Oxide (SiO_2).

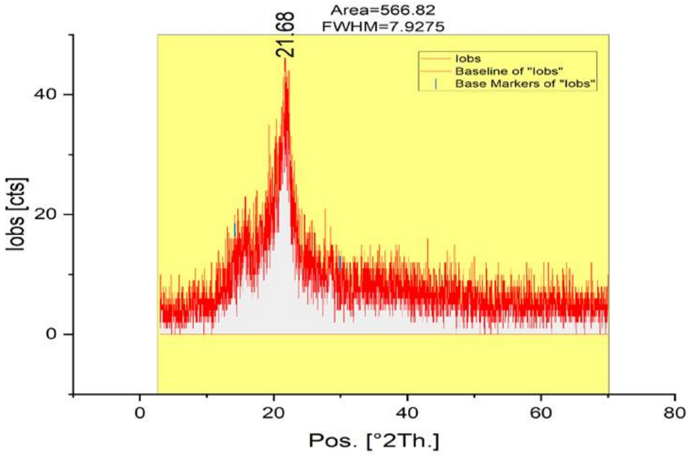

Figure 9: XRD of the composite before decomposition is done

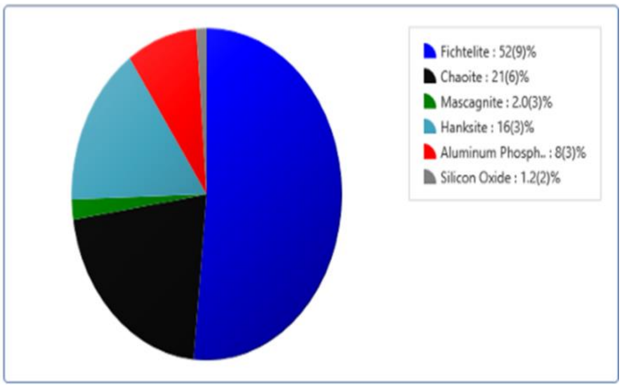

Figure 10: Phases present in the original composite

Figure 11 shows the XRD of the periwinkle reinforced polyester composite after decomposition has taken place. It is observed that the nitric acid treatment resulted in a shift in the major diffraction peak to 2θ = 25.86° and a 50.72% rise in CI, indicating higher crystalline order. Figure 12 shows the phases present in the decomposed polyester composite. New phases, including Flagstaffite (Sb_2O_3), Marialite ($Na_4CaAl_3Si_9O_{24}Cl$), Taenite (Fe-Ni), and Colusite ($Cu_{26}As_2S_{17}$), have emerged. These phase changes show that the process promoted the rearrangement of inorganic residues into crystalline forms that are thermodynamically stable.

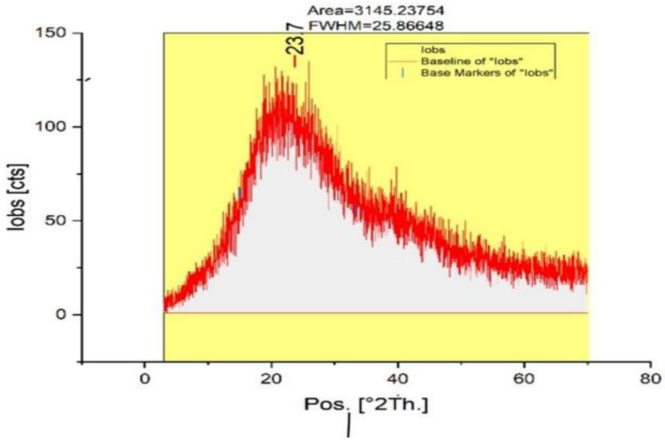

Figure 11: XRD of the composite after decomposition takes place

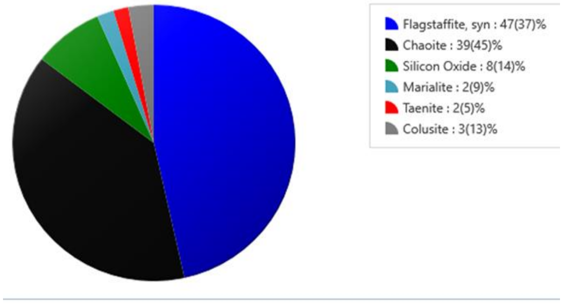

Figure 12: Phases present in the decomposed residue

3.4. Gel Chromatography-Mass Spectrometry (GC–MS) Analysis

Figure 13 shows the results of the Gel Chromatography – Mass Spectrometry analysis of the decomposed residue in order to clarify the organic components that were released by the polyester and the periwinkle shell. The Total Ion Chromatogram (TIC) showed the appearance of numerous peaks in the retention time interval of about 4.6 to 21.2 minutes as summarized in Table 1. This table portrays the development of a broad range of volatile and semi volatile degradation products.

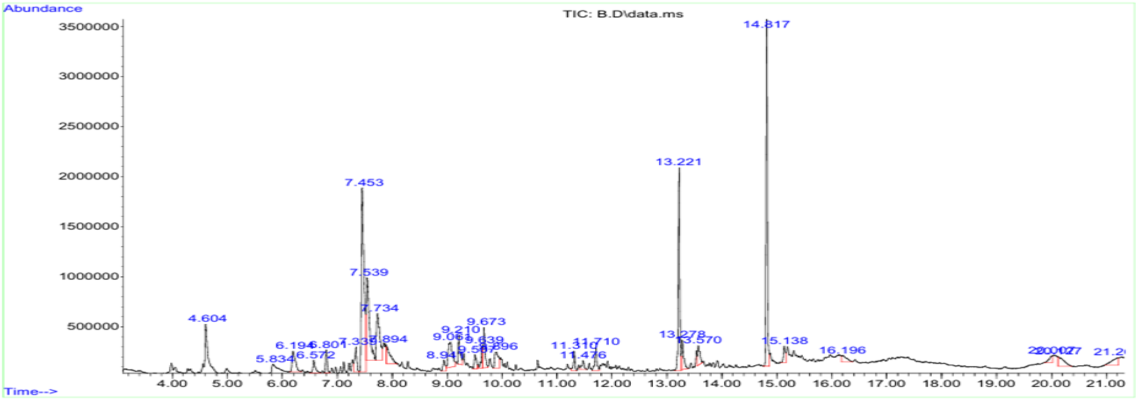

Figure 13: Gel Chromatography Mass Spectrometry (GC-MS) of the residue

Table 1: Summary table of identified compounds in the decomposed sample

Retention time (min)	Identified compound	Chemical Class
4.604	Benzoic acid, methyl ester	Aromatic ester (polyester degradation)
5.834	Benzoic acid	Aromatic acid
6.572	Thymoquinone	Quinone / oxygenated aromatic
6.801	Dodecane	Aliphatic hydrocarbon
7.453	Phthalic anhydride	Aromatic anhydride (polyester backbone)
7.539	Ninhydrin	Aromatic derivative
7.734	4-Nitrobenzaldehyde	Aromatic aldehyde
8.941	Dimethyl phthalate	Aromatic ester
9.061	Nitro-benzoic acid methyl ester	Aromatic ester
11.476	Methyl tetradecanoate	Fatty acid methyl ester
13.221	Hexadecanoic acid, methyl ester	Fatty acid methyl ester
13.570	n-Hexadecanoic acid	Fatty acid
14.817	Methyl stearate	Fatty acid methyl ester
15.138	Octadecanoic acid	Fatty acid
16.196	β-Sitosterol	Phytosterol (biogenic marker)
20.007	Stigmasterol	Phytosterol (biogenic marker)
20.127	Stigmasterol	Phytosterol (biogenic marker)

The large proportion of the compounds detected was made up of aromatic acids, aromatic esters and anhydrides such as benzoic acid, benzoic acid methyl ester, nitrobenzoate derivatives and phthalic anhydride. The presence of phthalic anhydride (17.83%) is important since it is a typical product of the degradation of unsaturated polyester resins that are typically made of phthalic or maleic anhydride (Dholakiya, 2012). A number of phenolic, aldehydic, and quinone-related compounds, including thymoquinone, nitrobenzaldehyde, and substituted phenols, were also discovered. These compounds could possibly be due to secondary degradation reactions of aromatic polyester fragments and organic matter that are related to the periwinkle shell. The periwinkle shell holds on to the leftover organic matrices, such as proteins and polysaccharides, that can produce phenolic and aromatic oxygenated compounds during the decomposition process. Long-chain aliphatic hydrocarbons (e.g., dodecane, heneicosane, pentacosane, heptacosane) were also present in the chromatogram. These compounds are said to be a result of disintegration of aliphatic parts of polyester resin, and organic matter that was incorporated in the shell structure. This type of hydrocarbons can be usually found in the degradation products of polymeric composites and biogenic fillers (Kigozi et al., 2016; Sarker et al., 2014).

The mid-to-late eluting compounds were composed of fatty acids and fatty acid methyl esters such as hexadecanoic acid methyl ester (10.01%), methyl stearate (15.40%), and fatty acid derivatives. The derivation of these species is probably due to lipid residues that were naturally present in the organic matter of periwinkle shells, or to the esterification reactions of chemical decomposition. Their presence also shows that, besides the synthetic polyester phase, the composite had a quantifiable quantity of bio-derived organic constituents. Phytosterols, including 2-sitosterol and stigmasterol, were observed at increased retention times. Though in lesser amounts, these compounds are excellent sources of biogenic material, the presence of which proves that organic materials related to the periwinkle shell were not fully destroyed in the process of composite manufacturing. The hybrid character of the composite, consisting of synthetic polymer and the components of natural shells, is emphasized by the persistence of the decomposition products.

In general, the GC -MS data shows that periwinkle shell polyester composite breaks down to a complex mixture of aromatic polyester-derived compounds, aliphatic hydrocarbons, fatty acids, and biogenic sterols. The prevalence of phthalic anhydride and aromatic esters proves the formation of the matrix by the polyester, and the detection of fatty acids and sterols proves the impact of the periwinkle shell filler.

3.5. Possible Chemical Reactions During Decomposition of Periwinkle-Reinforced Polyester Resin

3.5.1. Decomposition of the polyester resin (matrix)

The polyester matrix of the composite contains ester linkages (–COO–) in its backbone. When exposed to concentrated nitric acid (HNO_3), two main reactions took place: hydrolysis and oxidation.

a) Hydrolysis of ester bonds

The ester linkages were broken down by the acidic medium, forming carboxylic acid and alcohol groups. This reaction explains the emergence of sharper hydroxyl (–OH) peaks and stronger carbonyl (C=O) peaks in the FTIR spectra after decomposition.

Reaction is shown in Equation (3):

$$-COO- + H_2O \rightarrow -COOH + -OH \quad (3)$$

b) Oxidation of organic fragments

Nitric acid oxidized the resin's organic side chains and remaining styrene/benzene structures. This method generated carboxylic acids, aldehydes, and ketones, as validated by mass spectrometry. Nitric acid, a strong oxidizing agent, was converted to nitrogen oxides (NO_2), resulting in the observed brown color on the conical flask used.

3.5.2. Reaction of periwinkle shell (reinforcement: $CaCO_3$) with nitric acid

The periwinkle shell reinforcement consists mainly of calcium carbonate ($CaCO_3$). When treated with nitric acid, $CaCO_3$ dissolves, producing calcium nitrate, water, and carbon dioxide gas. The release of CO_2 contributed significantly to the observed weight loss of over 60%. Reaction is shown in Equation (4):

$$CaCO_3\,(s) + 2HNO_3\,(aq) \rightarrow Ca(NO_3)_2\,(aq) + CO_2\,(g) + H_2O\,(l) \quad (4)$$

3.5.3. Formation of nitrogen oxides (NO_2)

In addition to oxidizing the polymer, the decomposition process partially reduced nitric acid as shown in Equation (5). This produced nitrogen dioxide (NO_2), a brown gas that was noticed in the conical flask.

$$2HNO_3 \rightarrow H_2O + 2NO_2\,(g) + \tfrac{1}{2}O_2 \quad (5)$$

4. CONCLUSION

This experiment was able to show the chemical decomposition of periwinkle-reinforced polyester resin with nitric acid. The findings indicated that the rate of decomposition is highly dependent on acid concentration where 8M HNO_3 generated a total of 70 percent weight loss. FTIR was used to verify the presence of hydrolysis and oxidation of ester bonds, construction of hydroxyl and carbonyl groups, and stability of aromatic domains. XRD findings indicated growth in crystallinity index 45.36% to 50.72% with the emergence of new phases. The GC-MS results revealed the presence of leftover aromatic monomers, phthalates, and oxygenated species, which indicated the use of oxidative degradation. These findings confirm that nitric acid effectively decomposes the polyester matrix through oxidative and hydrolytic reactions, while simultaneously transforming the inorganic filler into stable crystalline products. The work contributes valuable knowledge to sustainable recycling of thermoset composites reinforced with natural fillers.

5. ACKNOWLEDGMENT

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6. CONFLICT OF INTEREST

There is no conflict of interest associated with this work.

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