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Thermal Characterization of Neem and Cork Wood Polyacrylonitrile Composites

By Dr. Mohammed Abid Ali & Dr. VVS. Kesava Rao
Lovely Professional University, India

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Dr. Mohammed Abid Ali [α] & Dr. VVS. Kesava Rao [σ]

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I. Introduction

Wood polymer composites (WPCs) results from the polymerization of liquid monomers already impregnated in wood. In principle WPCs should display super mechanical properties; dimensional stability to chemical degradation and less moisture absorb temperature than non-impregnated wood. A number of wood preservatives developed during those wood treatment processes and are under continuous demands which can develop the modified wood materials with improved mechanical strength, thermo-oxidative stability and resistance biodegradation for the better outdoor applications. Polymerization of polyacrylonitrile into poplar wood has also been reported and the composites indicated excellent moisture resistance and thermo oxidative stability [1, 2]. Temperature affects physical, structural properties of wood. Several affects have been made to establish the relationship between temperature and thermal stability of wood [3, 4, 5, 6]. The physical and mechanical properties of wood may be improved by preparing composites of wood with vinyl monomers [7]. Reinforcement of several monomers like styrene, methyl methacrylate has provided substantial thermal stabilities to different types of woods [8, 9]. However, since most vinyl monomers are non-polar; there is little interaction between these monomers and hydroxyl groups of the cellulose fibers. Wood, a renewable resource and naturally occurring material abundantly available has a wide range of applications as construction material, pulp, paper, fire board products as well as source of energy and as raw materials for various industrially important chemicals. Considerable work has been done on the modification of wood [10], [11]. Meyer (1981) reported that wood treated with vinyl type monomer followed by curing (radiation or catalyst) significantly improves the moisture resistance, hardness etc. The advantage of impregnation at normal conditions is the large quantities of samples of various sizes and shapes can be conveniently impregnated compared to vacuum impregnation [9]. Thermo gravimetric analysis (TGA) is one of the major thermal analysis techniques used to study the thermal behavior of carbonaceous materials. The rate of weight loss of the sample as a function of temperature and time is measured to predict thermal behavior of the materials. Thermal analysis as TG has become the polymer characterization method the most frequently used. The TGA is particularly more adopted for mass variation study. In this work, we studied the process of degradation of wood poly acrylonitrle composites. Compressive strength of impregnated eucalyptus wood specimens is greater than that of non impregnated ones indicating that monocomponent polyurethane resin can be considered for impregnated impregnating wood [12]. In thermo gravimetric list thermal decompositions of rice husk floor from room temperature to 350^{0}C was similar to that of wood floor. Thus rice husk floor was thought to be a substitute for wood floor in agricultural lignocellulosicfiber-thermoplastic composites in the aspect of thermal decomposition [13]. Physical and mechanical grown A auriculiformis of three different ages (8,12,13 years) from sirsi, Karnataka indicate that the wood can be used for tool handles in workshops and factories and agricultural sectors, light packing cases[14].

Author α: Associate Professor, Mechanical Engineering Department, K L University, Guntur, A. P, India. e-mail: mdabid_786@yahoo.co.in
Author σ: Professor, Department of Mechanical Engineering, AU College of Engineering Visakhapatnam–530003, A. P, India.

The mechanical stability of cedar wood samples were increased by using P (AGE/AN), P (AGE/MMA) copolymers. [15]. Polymerization of polymethyl methacrylate and acrylonitite into Block Berry Wood has also been reported and composites indicated excellent wear stability and thermo oxidation stability [16]. Polymerization of acrlonitrile into Indian Cork wood has also been reported and composites indicated excellent compressive resistance and thermal stability[17].

II. Experimental Procedure

a) Materials

All the chemicals and solvents (AR) were purchased from M/S SDFCL Chemicals Ltd; Mumbai. The monomer acrylonitrile was purified by extracting it with aqueous NAOH (10%) to remove inhibitor contents fallowed by repeated washings with distilled water. The fraction at 78^0 C was used for the impregnation polymerization reaction. Other chemicals and solvents were used without further purification.

b) Sample Preparation

Wood specimens were prepared as per IS: 1708-1986.The moisture content of wood was deduced according to ASTMD1037-72a and was found to be 12.75%.

c) Impregnation Procedure

The Benzene solution of acrylonitrile at concentration of 4M, 6Moles and Benzene solution of benzoyl peroxide at0.05M have also been prepared. Samples were then placed into an impregnation chamber. Extra loads were applied on the samples before impregnation so that no flotation occurs. The appropriate monomer system was then introduced through a dropping funnel and the specimens were left immersed while atmospheric pressure was reached and allowed to stand for up to 24H (ASTMD-1413-61). Treated wood specimens were then wrapped in commercially available Al foil and cured in oven at 95^0C for 2H to induce the impregnation polymerization reaction. Impregnation of polyacrylonitrile into neem, and cork woods was confirmed through scanning electron microscopy.

Figure 1 : Polymerization Process

III. Measurement of Loss of Mass

a) Charecterization Method

The DTG-TGA- DTA examines the process of weight changes as a function of time and temperature and other environmental conditions that may be created that may be created with in the instrument. In its simplest form, TGA measurements are made by heating the sample at constant rate in a prescribed atmosphere. W=f (Tor t). Thermo gravimetric analysis of polymers and their composites were carried out to assess the thermal and oxidation stability of the samples. The equipment specifications are: Model: TG/DTA6200. Temperature range: 5-600^0 C heating rate: 10^0C/min. The thermal stability of wood and related WPCs are usually measured through simultaneous Differential thermo gravimetry-Thermogravimetry-differential thermal analysis (DTG-TG-DTA).

b) Scanning Electron Microscope (SEM)

Electron micrographs of Cork wood &their Polyacrolonitrile (PAN) reinforced wood composites were scanned on HITACHI 3400N SEM. The morphologies of composites were studied in view to get a clear understanding about the affinity of polyacrylonitrile (PAN) with their respective woods.

IV. Results and Discussion

a) Thermal Analysis of Neem wood poly acrylonitrile composites

TG/DTA model thermal analyzer has been employed to study thermo gravimetry analysis of untreated wood and its polyacrylonitrile (PAN) wood composites in the atmosphere of nitrogen. 5 mg-20 mg masses of samples were analyzed. The sample was placed in a little cup made of aluminum hanging from a micro balance. The variation of the mass of the sample cans allows drawing the TG thermo grams. TG scans were exploited to evaluate the range for various decomposition stages electron micro graphs of woods and their reinforced wood composites were scanned on HITACHI 3400N SEM. TG data has been used to study the weight loss in Neem wood and related composites at the various temperatures range 0-600^{o}C. TG profile indicate the Thermo gravimetric analysis of wood started at 255^{o}C with –2.6% weight loss. A weight loss in Neem wood was recorded in temperature range 255^{o}C-314^{o}C with 15.7% weight loss which was further intensified at 383^{o}C with 37.4 % weight loss recorded. The first and second DTA endotherms have represented the crystallization temperature T_c at 81.7^{o}C and oxidation temperature T_{ox} at 371.7^{o}C. Similarly the maximum T_{max} and final T_f at decomposition were recorded 79.4^{o}C and 361.4^{o}C respectively from DTG endotherms. Thermo gravimetric analysis of Acrylonitrile impregnated wood composites with concentration of 2M started at 255^{o}C with 2.9% weight loss. A weight loss in Neem wood was

recorded in temperature range 255°C-314°C with 14.4% weight loss which was further intensified at 383°C with 45.9% weight loss recorded. The first and second DTA endotherms have represented the crystallization temperature T_c at 80.01°C and oxidation temperature T_{ox} at402.8°C .Similarly the maximum T_{max} and final T_f at decomposition were recorded 77.6°C and 366.1°C respectively from DTG endotherms. Thermo gravimetric analysis of Acrlonitrile impregnated wood composites concentration of 4M started at 255°C with 2.3% weight loss. A weight loss in neem wood was recorded in temperature range 255°C-314°C with 15.4% weight loss which was further intensified at 383°C with 40.1% weight loss recorded. The first and second DTA endotherms have represented the crystallization temperature T_c at 72.9°C and oxidation temperature T_{ox} at 374.5°C .Similarly the maximum T_{max} and final T_f at decomposition were recorded 66°C and 361.7°C respectively from DTG endotherms. Thermo gravimetric analysis of Acrlonitrile impregnated wood composites concentration of 6M started at 255°C with 3% weight loss. A weight loss in Neem wood was recorded in temperature range 255°C-314°C with 16.4% weight loss which was further intensified at 383°C with 34.9% weight loss recorded. The first and second DTA endotherms have represented the crystallization temperature T_c at88.2°C and oxidation temperature T_{ox} at 352.5°C .Similarly the maximum T_{max} and final T_f at decomposition were recorded 86.7°C and344.9°C respectively from DTG endotherms.

TG-DTG-DTA-scans were exploited to evaluate the ranges for various decomposition stages. (Graph Fig. 2 to Fig. 13).

b) Thermal Analysis of cork wood poly acrylonitrile composites

TG/DTA model thermal analyzer has been employed to study thermogravimetry analysis of untreated wood and its polyacrylonitrile (PAN) wood composites in the atmosphere of nitrogen. 5 mg-20 mg masses of samples were analyzed. The sample was placed in a little cup made of aluminum hanging from a micro balance. The variation of the mass of the sample cans allows drawing the TG thermograms. TG scans were exploited to evaluate the range for various decomposition stages electron micro graphs of woods and their reinforced wood composites were scanned on HITACHI 3400N SEM. TG data has been used to study the weight loss in cork wood and related composites at the various temperatures range 0-600°C. TG profile indicate the Thermo gravimetric analysis of wood started at 255°C with 1.4% weight loss. A weight loss in cork wood was recorded in temperature range 255°C-314°C with 16.8% weight loss which was further intensified at 383°C with 36.7 % weight loss recorded. The first and second DTA endotherms have represented the crystallization temperature T_c at 71.4°C and oxidation temperature T_{ox} at 393.3°C. Similarly the maximum T_{max} and final T_f at decomposition were recorded 70°C and 339.9°C respectively from DTG endotherms. Thermo gravimetric analysis of Acrylonitrile impregnated wood composites with concentration of 2M started at 255°C with 2.2% weight loss. A weight loss in cork wood was recorded in temperature range 255°C-314°C with 15.0% weight loss which was further intensified at 383°C with 41.9% weight loss recorded. The first and second DTA endotherms have represented the crystallization temperature T_c at 77.1°C and oxidation temperature T_{ox} at 372.3°C .Similarly the maximum T_{max} and final T_f at decomposition were recorded 74.5°C and 353.6°C respectively from DTG endotherms. Thermo gravimetric analysis of Acrlonitrile impregnated wood composites concentration of 4M started at 255°C with 2.0% weight loss. A weight loss in Cork wood was recorded in temperature range 255°C-314°C with 16.1% weight loss which was further intensified at 383°C with 36.4% weight loss recorded. The first and second DTA endotherms have represented the crystallization temperature T_c at 82.4°C and oxidation temperature T_{ox} at 402.9°C .Similarly the maximum T_{max} and final T_f at decomposition were recorded 77.3°C and 349.3°C respectively from DTG endotherms. Thermo gravimetric analysis of Acrlonitrile impregnated wood composites concentration of 6M started at 255°C with 2% weight loss. A weight loss in cork wood was recorded in temperature range 255°C-314°C with 15.7% weight loss which was further intensified at 383°C with 37.5% weight loss recorded. The first and second DTA endo therms have represented the crystallization temperature T_c at 78.2°C and oxidation temperature T_{ox} at 400.1°C. Similarly the maximum T_{max} and final T_f at decomposition were recorded 73.2°C and 351.8°C respectively from DTG endotherms.TG-DTG-DTA-scans were exploited to evaluate the ranges for various decomposition stages. (Graph Fig.14 to Fig.10).

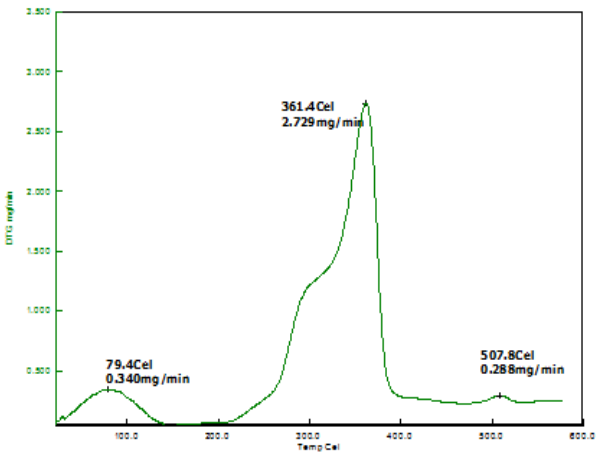

Figure 2 : D TG for Untreated neem wood

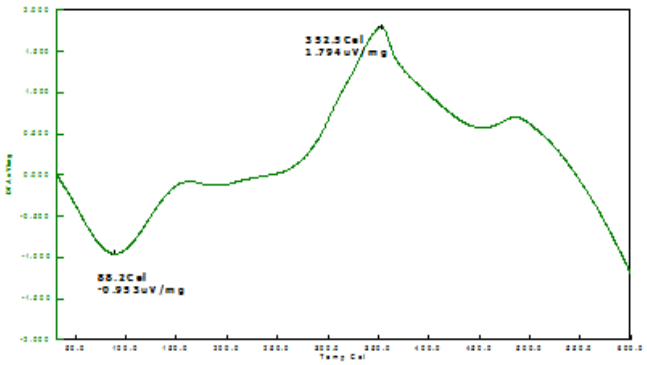

Figure 3 : D TA for Untreated neem wood

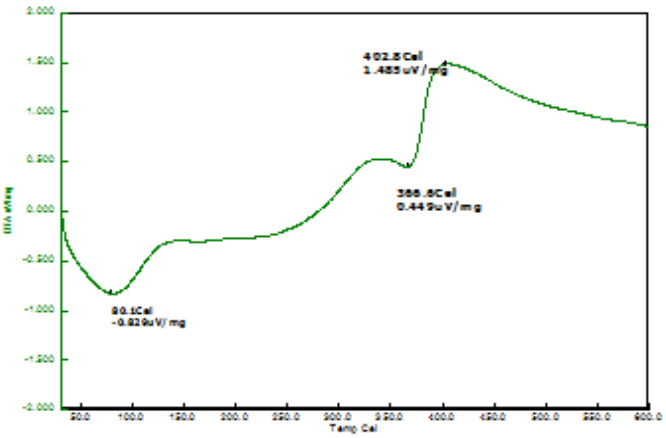

Figure 4 : TGA for Untreated neem wood

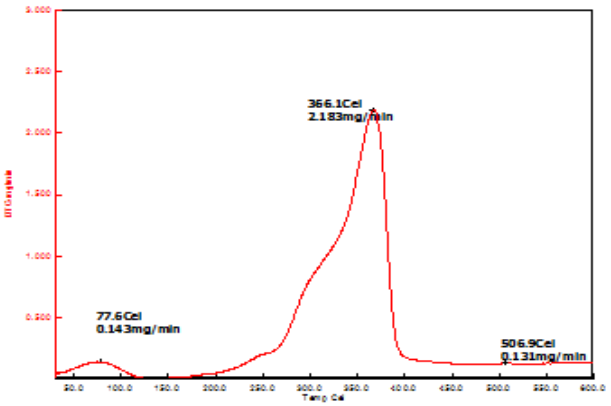

Figure 5 : DTG curve for Acrylonitrile of 2 Mconcentrated treated neem wood

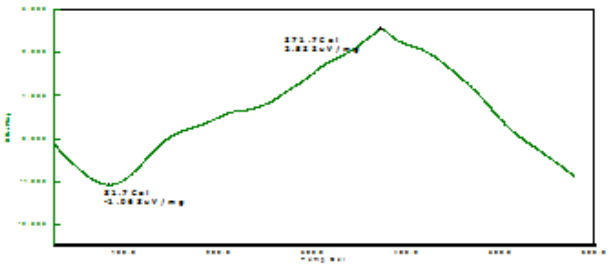

Figure 6 : DTA curve for Acrylonitrile of 2 Mconcentrated treated neem wood

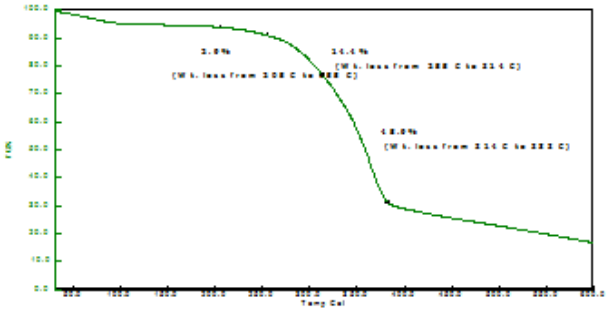

Figure 7 : TGA curve for Acrylonitrile of 2 Mconcentrated treated neem wood

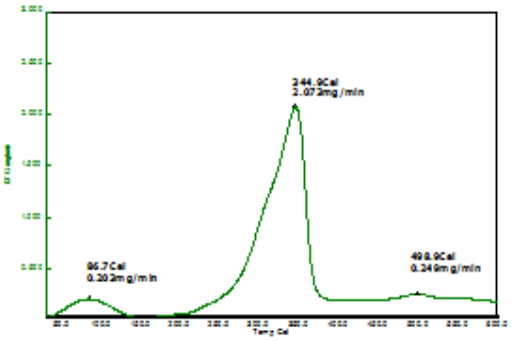

Figure 8 : DTG curve for Acrylonitrile of 4 Mconcentrated treated neem wood

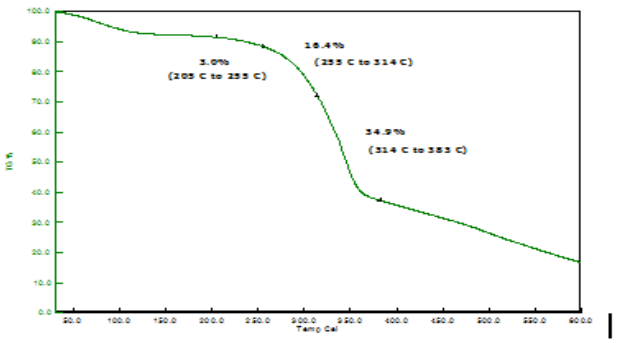

Figure 9 : TGA curve for Acrylonitrile of 4 Mconcentrated treated neem wood

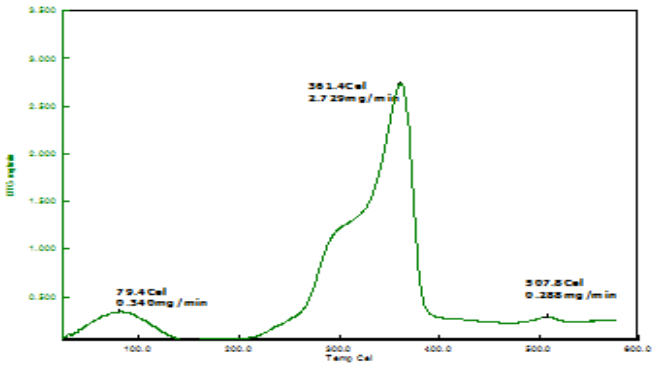

Figure 10 : DTA curve for Acrylonitrile of 4 Mconcentrated treated neem wood

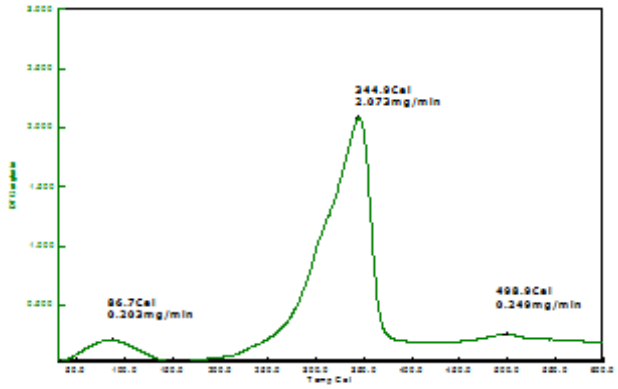

Figure 11 : DTG curve for Acrylonitrile of 6 Mconcentrated treated neem wood

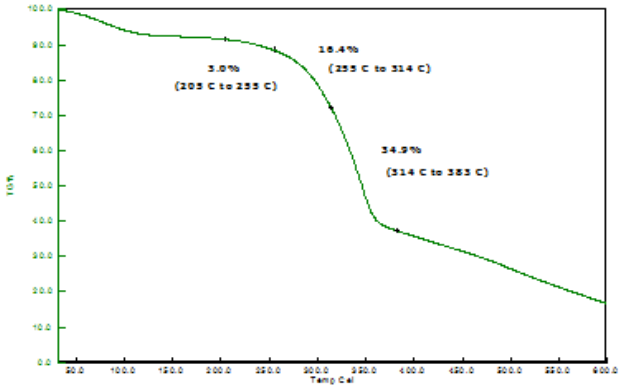

Figure 12 : TGA curve for Acrylonitrile of 6 Mconcentrated treated neem wood

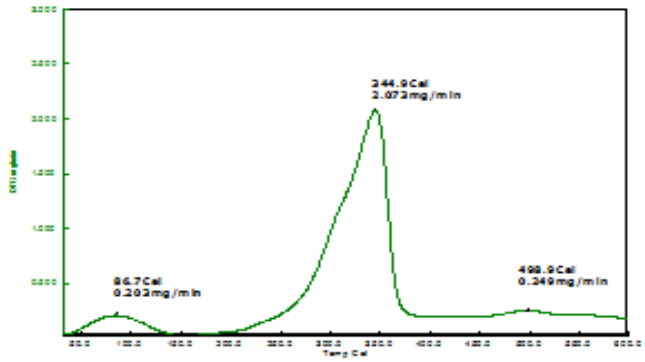

Figure 13 : DTG curve for Acrylonitrile of 6 Mconcentrated treated neem wood

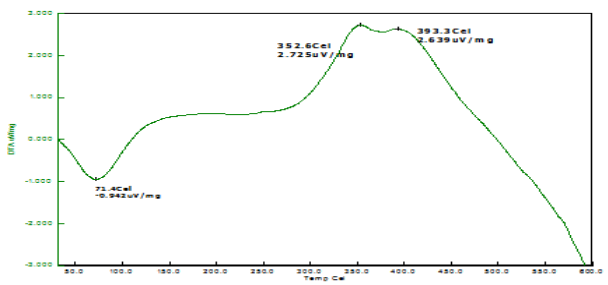

Figure 14 : DTA for Untreated cork wood

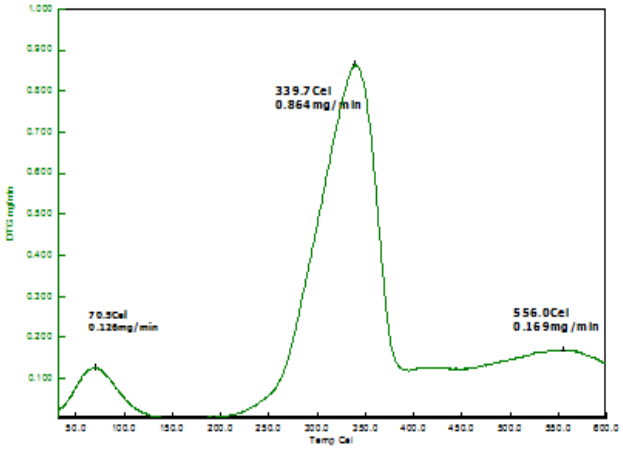

Figure 15 : D TG for Untreated cork wood

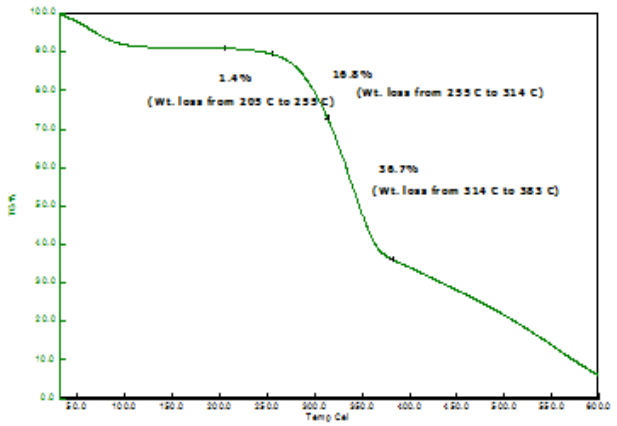

Figure 16 : TGA for Untreated cork wood

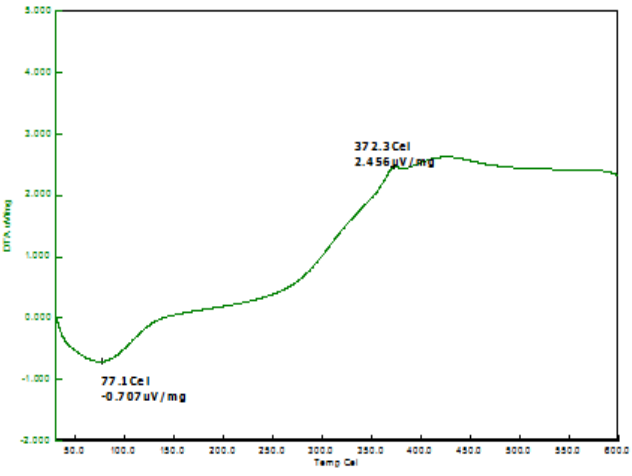

Figure 17 : DTA curve for Acrylonitrile of 2M concentrated treated cork wood

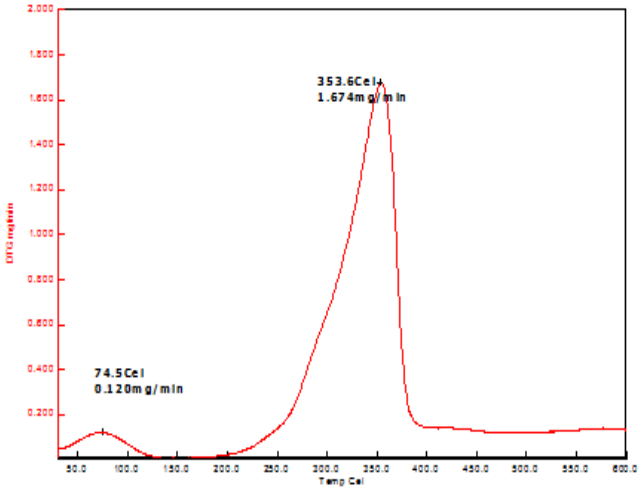

Figure 18 : DTG curve for Acrylonitrile of 2M concentrated treated cork wood

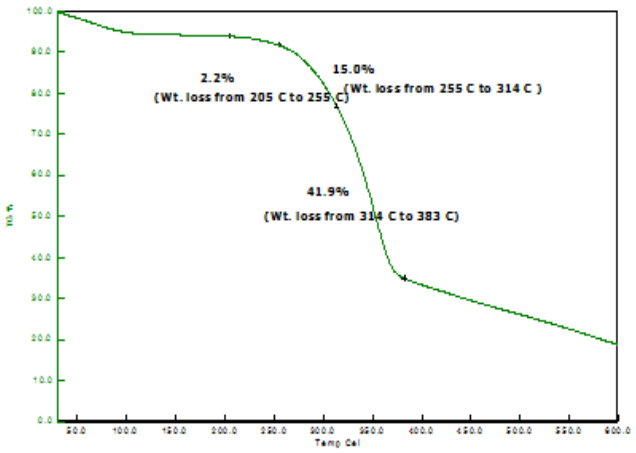

Figure 19 : TGA curve for Acrylonitrile of 2M concentratedtreated cork wood

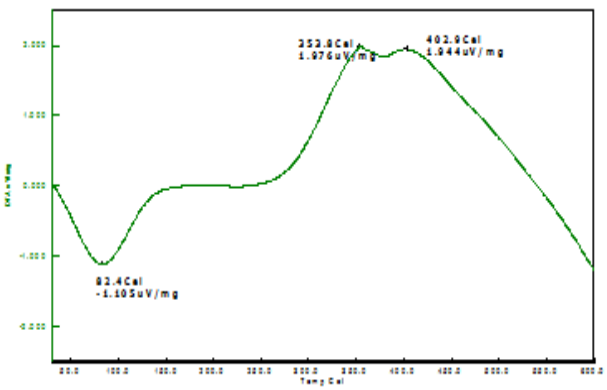

Figure 20 : DTA curve for Acrylonitrile of 4M concentrated treated cork wood

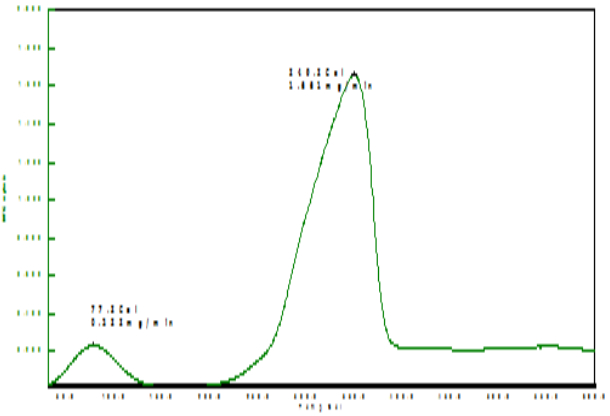

Figure 21 : DTG curve for Acrylonitrile of 4M concentrated treated cork wood

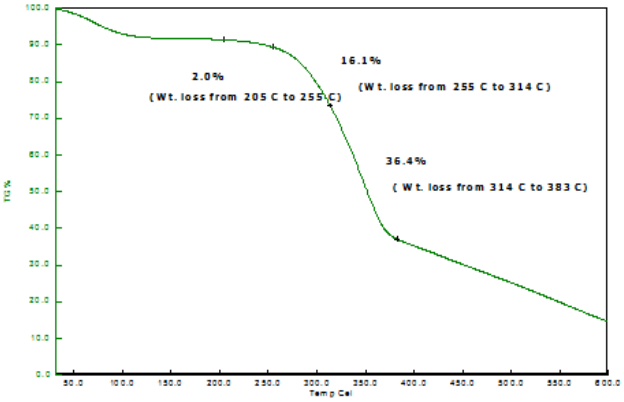

Figure 22 : TGA curve for Acrylonitrile of 4M concentrated treated cork wood

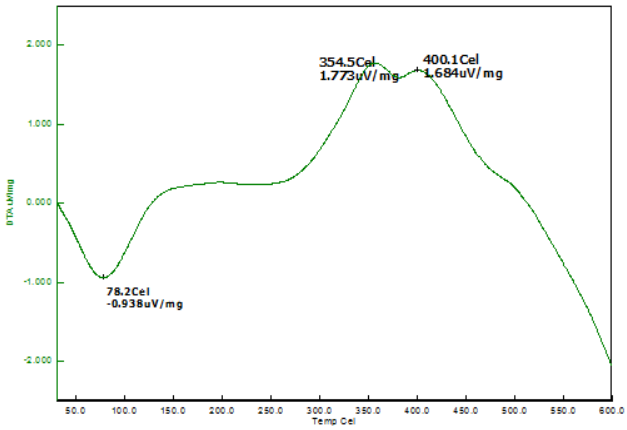

Figure 23 : DTA curve for Acrylonitrile of 6M concentrated treated cork wood

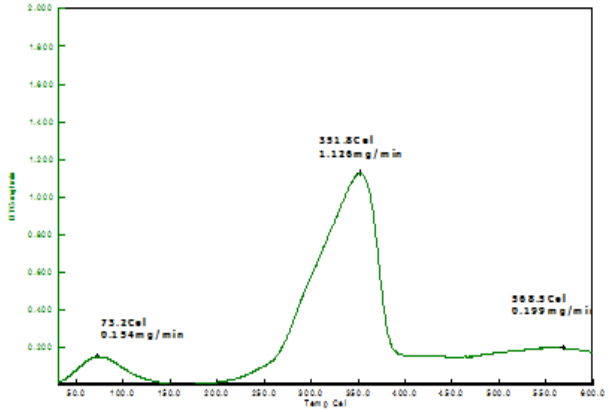

Figure 24 : DTG curve for Acrylonitrile of 6M concentrated treated cork wood

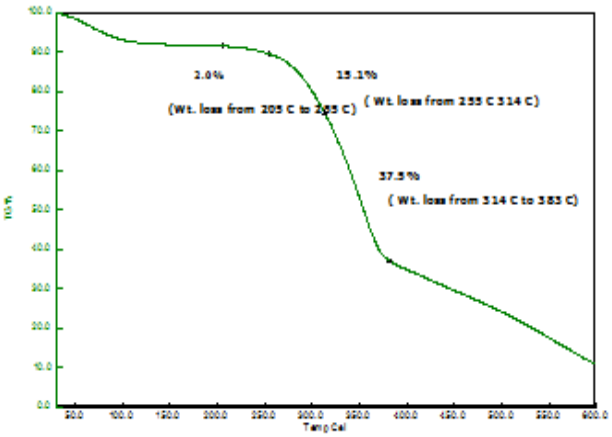

Figure 25 : TGA curve for Acrylonitrile of 6M concentrated treated cork woo

c) Scanning Electron Microscope (SEM)

SEM test is a type of electron microscope that images the sample surface by scanning it with a high-energy beam of electrons in a raster scan pattern. The electrons interact with the atoms that make up the sample producing signals that contain information about the sample's surface topography. Scanning electron microscopic analysis cork wood specimens examined the surface topology of untreated and treated Wood specimens. The pore size of the material was continuously affected by increasing the extent of Acrylonitrile reinforcement. The analysis of microscopic structure of non-impregnated cork wood samples compared with the samples impregnated with Poly Acrylonitrile monomers shown in Fig. 26 to Fig. 33.

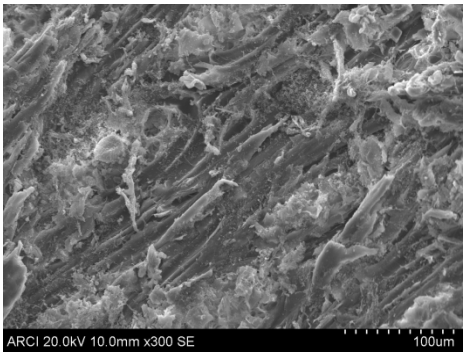

Figure 26 : Microscopic view of untreated neem wood

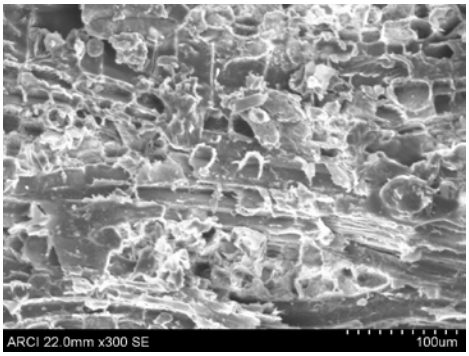

Figure 27 : Microscopic view poly acrylonitrile affinity

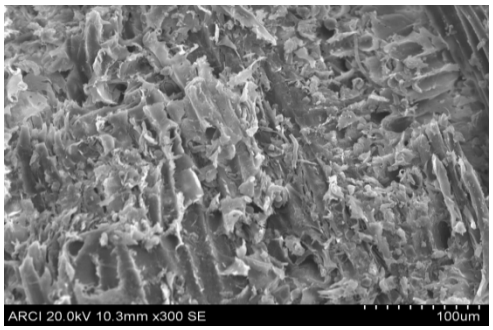

Figure 28 : Microscopic view poly acrylonitrile

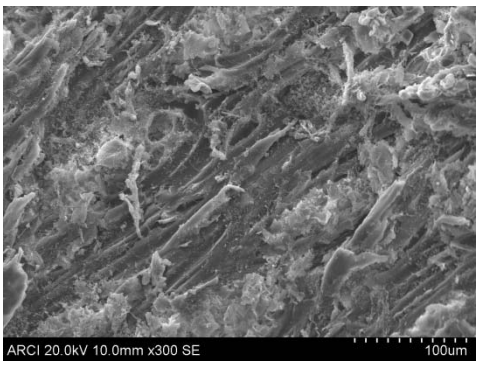

Figure 29 : Microscopic view poly acrylonitrile affinity of 6M concentration treated neem wood

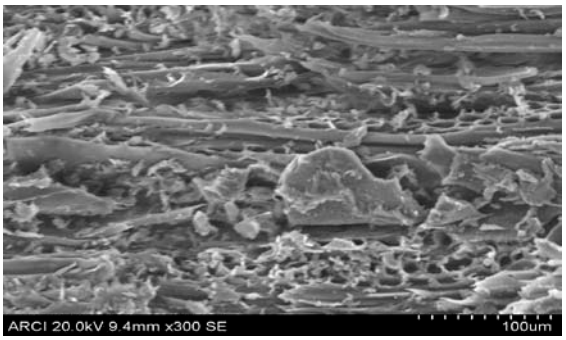

Figure 30 : Microscopic view of untreated cork wood

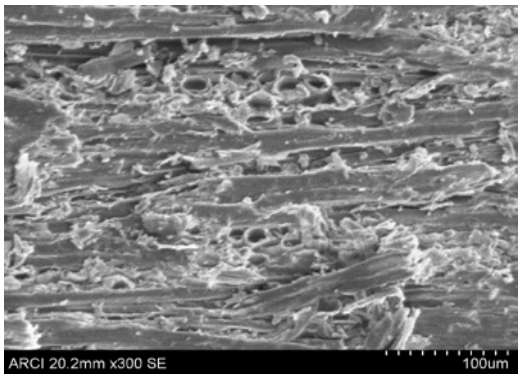

Figure 31 : Microscopic view poly acrylonitrile affinity of 2M concentration treated neem wood

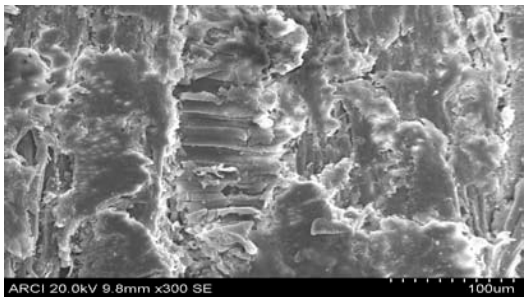

Figure 32 : Microscopic view poly acrylonitrile affinity of 4M concentration treated neem wood

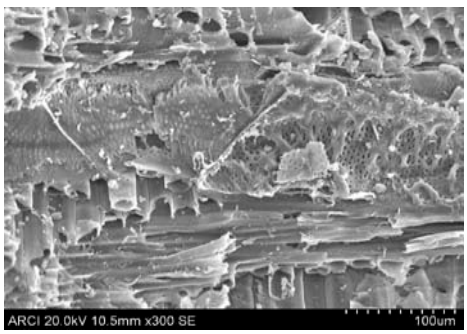

Figure 33 : Microscopic view poly acrylonitrile affinity of 6M concentration treated cork wood

V. Conclusions

Results of these tests demonstrated that the Thermal properties of impregnated wood specimens are greater than that non impregnated ones. It is concluded that thermal data presented in this paper that thermal stability of Polyacrylonitrile (PAN) Reinforced composites was improved in comparison to untreated neem and cork woods. Scanning microscope (SEM) indicated non-uniform distribution of polyacrylonitrile into wood lumens. It is concluded that observations that thermal stability of low grade woods can incorporated for its vertesile scientific and technological applications by selecting appropriate combinations of the monomer and catalyst along with their respective concentrations. Results of these tests demonstrated that the Thermal properties of impregnated wood specimens are greater than that non impregnated ones.

It is concluded that thermal data presented in this paper that thermal stability of Polyacrylonitrile (PAN) Reinforced composites was improved in comparison to untreated cork and neem woods. Scanning microscope (SEM) indicated non-uniform distribution of polyacrylonitrile into wood lumens.

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