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AMMONIUM AND CHOLINIUM BASED IONIC LIQUIDS AS A NEW CHEMICAL MODIFIER FOR GIANT BAMBOO (DENDROCALAMUS ASPER)-POLYPROPYLENE THERMOPLASTIC COMPOSITE PROCESSING

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Abstract

The purpose of this study is to determine the effect of chemical modification on giant bamboo (Dendrocalamus Asper) using ammonium and cholinium based Ionic Liquids on the structure, thermal, and mechanical properties of bamboo-polypropylene (PP) thermoplastic composites. Three new ionic liquids have been successfully synthesized and used for this purpose, which are triethylammonium hydrogen sulphate, benzyl triethylammonium acetate, and cholinium acetate. Quaternarization reactions and anion metathesis are used to synthesize these ionic liquids. The ionic liquids are characterized by their structure using Fourier Transform Infrared (FTIR) and Nuclear Magnetic Resonance (1H-NMR) Spectroscopy, and their thermal properties using Thermogravimetry-Differential Thermal Analyzer (TG/DTA). Bamboo treated ionic liquids is characterized using FTIR, TG/DTA, and X-Ray Diffraction (XRD). The success of bamboo modification is shown by the FTIR spectrum with the existence of new peaks and wavelength shifts of each bamboo modified with ionic liquids. Bamboo-polypropylene composites are made through a moulding process. Composites are characterized by their structure using FTIR and XRD, Scanning Electron Microscope (SEM), their thermal properties using TG/DTA, and their mechanical properties using tensile tests. Ionic liquid treatment on bamboo succeeded in reducing the crystallinity of PP as seen from the XRD peak with a percentage of PP crystallinity of 49.8%. The PP composites with unmodified bamboo fillers and bamboo with chemical modification of triethylammonium hydrogen sulphate, cholinium acetate, and benzyl-triethylammonium acetate are 47.7; 39.9; 48.6; and 49.3%. The interaction between polypropylene modified bamboo is seen from SEM, as well as the mechanical properties are shown by the tensile strength of PP, composites with unmodified bamboo fillers and fillers modified bamboo of triethylammonium hydrogen sulphate, cholinium acetate, and benzyl triethylammonium acetate are 0.44; 14.86; 16.16; and 14.31 MPa.

Keywords: Ammonium, Cholinium, Giant Bamboo (Dendrocalamus Asper), Ionic liquids, Polypropylene, Thermoplastic composites.

1. Introduction

In line with the need for reliable and environmentally friendly structural and construction materials, there is an even tremendous tendency to use thermoplastic composite materials for this purpose. Besides, research to engineering thermoplastic composites by utilizing natural components is also increasing [1]. Lignocellulosic biomass is an alternative source of primary composite fillers, because of its renewable nature. Lignocellulosic biomass usually has the main components of cellulose (30-50%), hemicellulose (20-35%), lignin (20-30%), and 2-6% by weight are other compounds [2]. The use of wood biomass in wood-polymer composites has been identified in various applications, but the increased use of wood will contribute to greater forest exploitation. One solution to this problem is to find a companion material and/or substitute wood as a composite filler such as bamboo.

Bamboo can grow and spread widely in tropical lands, such as Indonesia. Indonesia has 157 types of bamboo, 60-70 of which are typical and do not exist in other countries. There are around 1200-1300 bamboo species in the world and 11.9% of them are in Indonesia [3]. Bamboo is a plant that does not require special care, has a rapid growth rate (can reach 91.3 cm/day) and can be harvested with excellent quality as a structural and construction material at the age of 3-4 years.

Bamboo is a plant that is known to have high mechanical properties [4]. Bamboo has a higher compressive strength than wood, brick, or concrete, and a tensile strength surpassing steel [5]. Wang et al. (2014) found that bamboo fibre has strengths that are almost similar to glass fibre. Bamboo material, as a lignocellulose material with a strong backbone structure and a high strength/weight ratio, can be the filler of choice for thermoplastic composites. Lignocellulose also causes fewer scratches on processing machines than glass fibre.

In plastic composites, thermoplastic polymer materials, such as polypropylene (PP), act as matrices. Polypropylene has been used for various purposes such as plastic for food packaging, beverage packaging, and household appliances. In 1999, Badan Pusat Statistik (BPS) data shows the trade volume of Indonesia's imported PP plastics 1999 was 182,523.6 tons [6]. This polypropylene waste (like other plastic wastes) is an environmental problem that requires fast and comprehensive handling. Plastic waste disposed of each household on average reaches 9.3% of total household waste.

The main obstacle that often arises in the processing of composite materials is the aspect of achieving adherence and the interface between the polymer matrix and the lignocellulosic filler [7]. The unmodified lignocellulose component shows a tendency for aggression which is reflected by imperfect filler dispersion in the polymer matrix so deterioration of mechanical properties occurs [8].

This research is conducted to modify the surface of giant bamboo fibre on the processing of cellulose composites on giant bamboo fibre (Dendrocalamus Asper) with thermoplastic polymers of polypropylene waste using ionic liquids. Ionic liquids are materials that only consist of ionic species (cations and anions), do not contain certain neutral molecules, and have a relatively low melting point, located at temperatures <100-150ºC, although generally at room temperature [9]. In contrast to molten salt which usually has a high melting point and viscosity, it is also highly corrosive. Ionic liquids are generally liquid at room temperature, have relatively lower viscosity, and have relatively no corrosive properties [10]. Ionic liquids have a very wide liquid range; not volatile (non-volatile); non-flammable; high heat, chemical and electrochemical stability (in some cases have thermal stability up to 400ºC); negligible vapour pressure value; the ability to dissolve many organic and inorganic compounds; and miscibility which varies with water and organic solvents [11]. Ionic liquids have been used as solvents for various purposes of modifying cellulose. Acrylates, benzoylates, carbamates, and choline acetate derivatives from biopolymers have been successfully made in many ionic liquids [12-15]. Borysiak, et al. (2018) has carried out chemical modification on wood using ionic liquids in didecyldimethylammonium bis (trifluoromethyl sulfonyl) imides and used as fillers in wood-polypropylene composites, and the results showed a positive influence on the mechanical properties of the composite materials.

This study was inspired by the results of research by Borysiak, et al. (2018) and used the idea to modify the bamboo material as a filler in the polypropylene matrix so its effect on the supramolecular structure and mechanical properties of cellulose, polymers and composites can be studied. The chemical treatment of wood was conducted by Borysiak, et al. (2018) solely, for the first time, with newly synthesized ionic liquid, didecyldimethylammonium bis (trifluoromethyl sulfonyl) imide. In this study, ionic liquids that will be used as modifiers are cheap and easy to synthesized ionic liquids with three different cations, namely cholinium, hydrogen triethylammonium and benzyl-triethyl ammonium with hydrogen sulphate and acetic anions. These three types of cations are chosen because of their ability to penetrate the cellulose structure [16], while the hydrogen sulphate and acetate anions are chosen because of its high basicity, so it is expected to modify the cellulose structure in bamboo more efficiently [16].

From a scientific point of view, the study carried out will enrich the scientific field because it can gain knowledge about the effect of bamboo modification on the thermal and mechanical properties of composites. Indonesia is a country with a very large variety of bamboo. Plastic waste (such as polypropylene) has also become an environmental problem that needs to be anticipated. This study provides a new perspective on how bamboo and plastic waste can be used in Indonesia. The bamboo and plastic waste processing methods offered in this study are practical so everyone can do it.

2. Research Method

2.1. Materials and characterization methods

The materials used in this study are giant bamboo (Dendrocalamus Asper). The bamboo is 7-year-old bamboo taken from an area in Subang, West Java. Polypropylene is taken as waste from mineral water packaging glass. Triethylamine p.a. (Merck), benzyl chloride p.a. (Merck), choline chloride p.a (Aldrich), 95-97% sulfuric acid (Merck), sodium acetate p.a. (Merck), acetone p.a. (Merck), and ethanol p.a. (Merck) are used without prior purification. The equipment used in this study is a ball mill/shaker mill (HEM-E3D), hot plate (DLAB MS-H280-Pro, IKA C-MAG HS7), rotatory evaporator (BUCHI), and 140 mesh test sieves (KPK Product). Analytical instruments used for structure characterization are FTIR (Shimadzu Prestige 21), and 1H-NMR. For the analysis of physicochemical properties, the TG/DTA Analyzer (HITACHI-720), XRD (Bruker D8 Advance) and SEM (Hitachi-SU3500) are used. For the mechanical test, the Strength Tester (Tensolab-5000) is used.

2.2. Synthesis of ionic liquids

Triethylammonium hydrogen sulphate ionic liquid can be easily synthesized through the acid-base neutralization reaction of the triethylamine reagent (10.1 g, 1 mol) with 18 M sulfuric acid (9.8 g, 1 mol). Triethylamine is mixed with sulfuric acid dropwise while stirring for an hour at a temperature of 60-70˚C. The solution is then dried to remove the solvent using an evaporator at 90ºC until a solid white is obtained. Ionic liquids resulting from synthesis are then characterized by FTIR, 1H-NMR and TG/DTA.

The synthesis of benzyl-triethyl-ammonium acetate is performed by mixing 1 mole of benzyl-triethyl-ammonium chloride with 1 mole of sodium acetate and dissolving it into acetone. The mixture is stirred (100 rpm) and heated (T = 50-60 ºC). After an hour the mixture is filtered to obtain solid residues and filtrate. The filtrate is then dried using a rotatory evaporator until a solvent-free solid is obtained. Benzyl-triethyl-ammonium chloride is synthesized through the quaternization reaction of benzyl-chloride (1 mol) and triethylamine (1 mol). Triethylamine is added to benzyl-chloride by dropping, the mixture is stirred (130 rpm) and heated at a temperature of 50-60ºC for 2 hours. Then the mixture is allowed to stand for 24 hours at room temperature. The mixture is then evaporated using a rotatory evaporator for 10 hours. Benzyl-triethyl-ammonium chloride is a white solid and then characterized by FTIR, 1H-NMR and TG/DTA.

The Cholinium ionic liquid is synthesized through the chloride anion metathesis reaction of the cholinium chloride ($C_5H_{14}ClNO$) (10 g, 1 mol) with the acetate anion of sodium acetate (10 g, 1 mol) using acetone as the solvent. The mixture is heated at a temperature of 50-60ºC and stirred at 100 rpm for an hour. After that, the mixture is filtered until a solid residue and filtrate are obtained. The filtrate is then evaporated until a solvent-free solid is obtained and then characterized by FTIR, 1H-NMR and TG/DTA.

2.3. Preparation of bamboo powder

Bamboo is logged under the minimum humidity conditions. Logging is conducted during the day. The top third of the cut is taken and cut into small pieces 1 cm^3 in size. These small pieces are oven-dried at 70ºC for 24 hours and pulverized in a ball mill for five hours. The fine bamboo powder is then dried again in a 70ºC oven for 12 hours. The fine powder is then filtered using a 140-mesh sieve test. Powder size is also determined using SEM.

2.4. Chemical modification of bamboo using ionic liquids

Each ionic liquid is dissolved into ethanol (0.2:1). The bamboo powder is then put into each ionic liquid solution in ethanol by stirring (100 rpm) and heated at 70ºC for three hours. The mixture is then filtered, and the bamboo residue is washed using ethanol three times. Then, the bamboo is dried in the oven (T = 70ºC). The modified bamboo is then characterized using FTIR and TG/DTA.

2.5. Bamboo-polypropylene composites processing

Composite processing is conducted using the moulding method, where polypropylene is heated at a temperature of 170ºC, after the polypropylene is completely melted, the bamboo is added then stirred using a mechanical stirrer with a speed of 2500 rpm. After homogeneous, the mixture is printed using hot-press with a pressure of 3000 Pascal. Bamboo-polypropylene composites are characterized using FTIR, TG/DTA, XRD, SEM, and Tensile Test.

3. Results and Discussions

3.1. Characterization of modified bamboo

The FTIR spectrum shows changes from bamboo before modification and after modification. Modified bamboo gives a change that is indicated by the presence of a new peak. Compared to bamboo without modification, bamboo with the treatment of triethylammonium hydrogen sulphate ionic liquid, Fig. 1(b), gives a new peak at wave number 1110.99 cm^{-1} which is a CN bond, and at wave number 619.14 cm^{-1} is a typical CH of triethylammonium.

In modified bamboo using ionic liquid benzyl-triethyl-ammonium acetate, Fig. 1(c) shows a new peak at wave number 1107.14 cm^{-1} which is a CN bond, wave number 705.95 cm^{-1}, 752.23 cm^{-1}, 788, 88 cm^{-1} which is an aromatic CH, and at wavenumber 613.36 cm^{-1} which is a typical CH of triethylammonium.

Furthermore, in the modified cholinium acetate bamboo, Fig. 1(d), there is also a new peak at wave number 1107.14 cm^{-1} which is a CN bond, and at wave number 613.36 cm^{-1} which is a typical CH bond [1, 17-19].

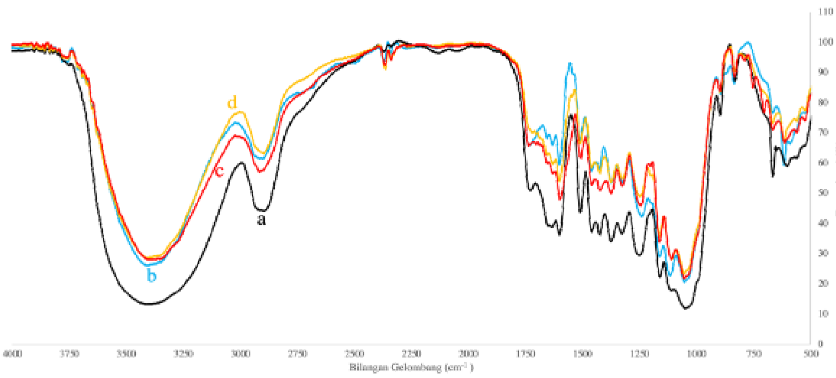

Fig. 1. FTIR Spectrum without modified bamboo and modified bamboo ionic liquids (500-4000 cm-1): (a) unmodified bamboo (b) bamboo-triethyl-ammonium hydrogen sulphate (c) bamboo-benzyl-triethyl-ammonium acetate (d) bamboo-cholinium acetate.

In addition to the emergence of new peaks, the success of modifying bamboo using ionic liquids is shown by the shifting of the wavenumber from -O-H as can be seen in Fig. 1, wherein non-modified bamboo there is a peak at wavenumber 3415.93 cm^{-1} which is free -O-H. In the modified bamboo, the OH peak shifted, where for the modified bamboo triethylammonium hydrogen sulphate shifted to wave number 3396.64 cm^{-1}, the modified bamboo benzyl-triethylammonium acetate shifted to the wavenumber 3344.57 cm^{-1}, and for the modified bamboo the acetate shifts to wave number 3406.29 cm^{-1}.

OH shift is related to the interaction between -O-H groups in bamboo cellulose with cations and anions of ionic liquid compounds [1]. The shift towards smaller wavenumbers is the result of interactions that produce ever-increasing hydrogen bonds. The bond energy values are presented in Table 1.

Table 1. Comparison of OH wavenumber friction and hydrogen bonding energy.

Sample	Wavenumber (cm^{-1})	E_H
Original Bamboo (Free O-H)	3415.93	-
Bamboo-[TEA]$^+$ [HSO4]$^-$	3396.64	1.48
Bamboo-[Benzil-TEA] $^+$ [CH_3COO]$^-$	3344.57	5.48
Bamboo-[Cholinium]$^+$ [CH3COO]$^-$	3406.29	0.74

Based on FTIR analysis, the chemical interaction between bamboo and ionic liquids can be predicted. The interaction that occurs is the interaction between the hydroxyl (OH) group of bamboo and groups of ionic liquids. In bamboo modified with triethyl-ammonium hydrogen sulphate ionic liquid, interactions occur between the hydroxyl group and the ammonium group in the ionic liquid, illustrated in Fig. 2.

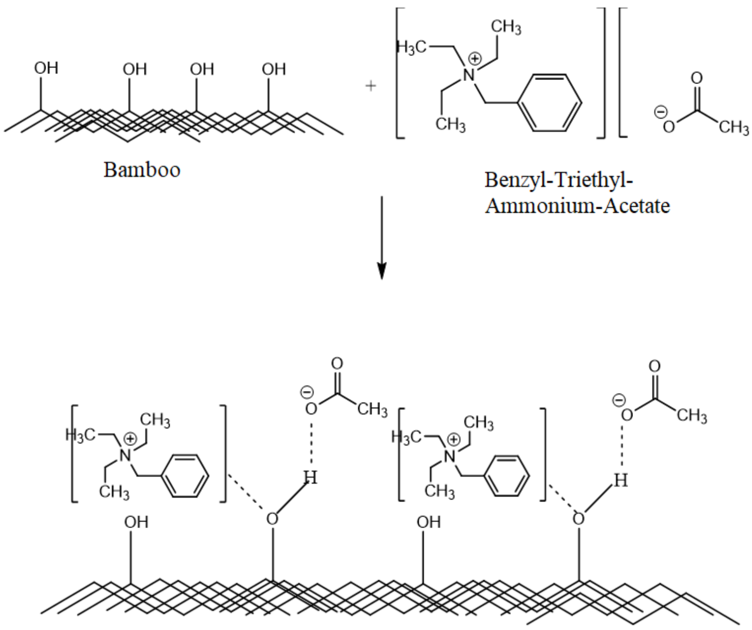

Fig. 2. Interaction of -O-H-Groups with triethyl-ammonium hydrogen sulphate ionic liquids.

In bamboo with a modification of benzyl-triethylammonium acetate liquid, interactions also occur between the hydroxyl group on bamboo and the ammonium group on ionic liquid. This interaction is illustrated in Fig. 3.

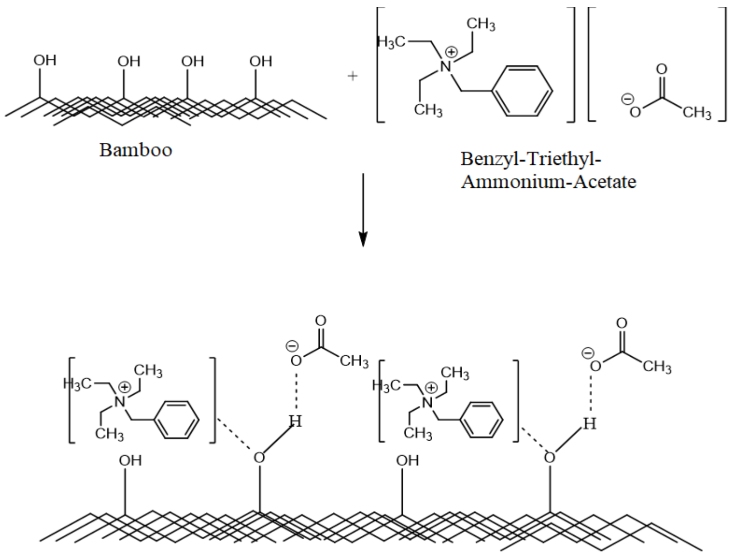

Fig. 3. Interaction of -O-H Groups in bamboo with benzyl-triethylammonium acetate ionic liquids.

For bamboo modified using ionic cholinium acetate liquid, the interaction between the hydroxyl group and the ammonium group in the ionic liquid is illustrated in Fig. 4.

The FTIR results indicate chemical interaction between wood and ionic liquids. The proposed interaction between hydroxyl groups of wood and the ammonium, cholinium, and acetate groups from the ionic liquid occurring during the modification process is illustrated in Fig. 2-4 [1, 17-18].

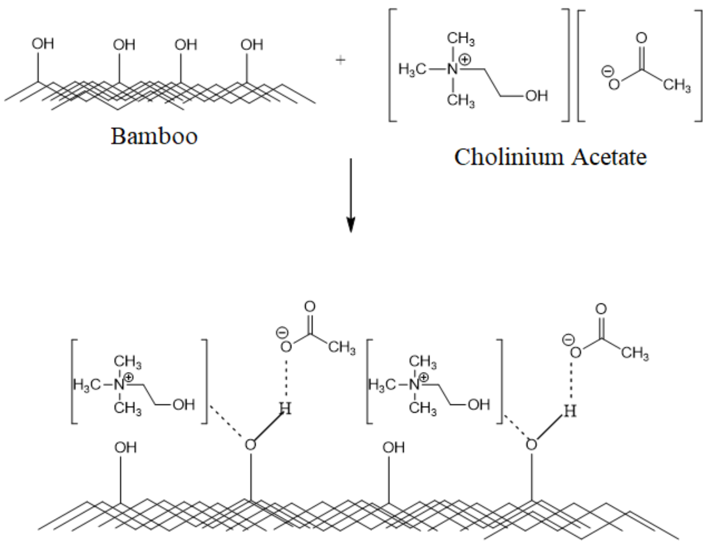

Fig. 4. Interaction of -O-H groups in bamboo with cholinium acetate ionic liquids.

The peaks in Fig. 5 that appear at 2θ = 15º, 17º and 22º are polymorphic cellulose I [1]. Based on the calculation of the crystalline and amorphous peak area, the crystallinity of the non-modified bamboo and modified bamboo with ionic liquid triethylammonium hydrogen sulphate, benzyl-triethylammonium acetate, and cholinium acetate are 16.3, 22.4, 14.0, and 16%, respectively. 3% of the decreasing degree of crystallinity of bamboo after adding benzyl-triethylammonium acetate ionic liquid is considered as a result of intra- and intermolecular hydrogen interactions in the cellulose region of bamboo. Where the influence of ionic liquid reaction on bamboo cellulose causes cellulose chain mobility, resulting in a decrease in crystallinity [1].

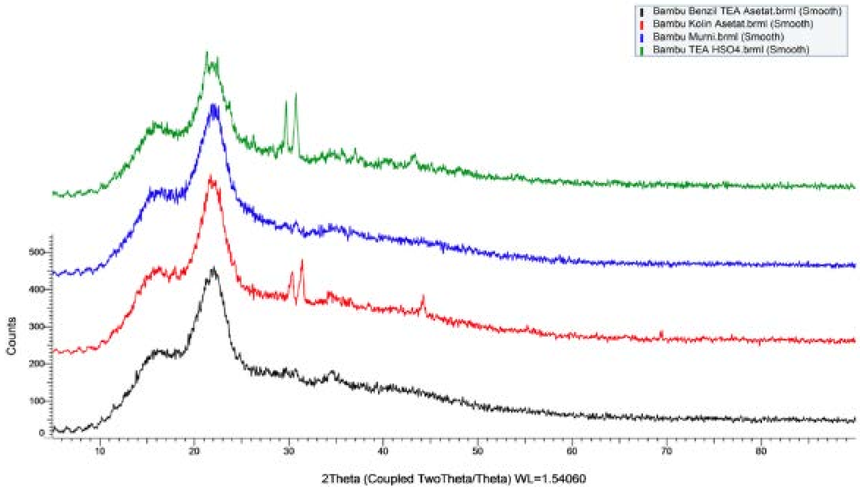

Fig. 5. Modified bamboo x-ray diffraction pattern and bamboo without modification.

3.2. Characterization of bamboo-polypropylene composites

In the analysis using TG/DTA, thermal stability and decomposition point of the bamboo-PP composite are known. This is conducted to adjust the use and application of composite products. In Fig. 6, it is shown that the decomposition point in composites with an ionic liquid-filled bamboo filler has increased temperature or in other words has increased thermal stability, compared to composites without modification.

The temperature of the composite decomposition point with pure bamboo fillers in Fig. 6 is 234˚C, bamboo-triethylammonium hydrogen sulphate fillers 242°C, bamboo-benzyl-triethylammonium acetate fillers 267°C, and bamboo-cholinium acetate fillers 276°C.

Based on the modified bamboo using ionic liquids, the crystallinity values are different. This certainly affects the composite crystallinity. The formation of this supramolecular composite structure depends on the processing conditions. The hexagonal shape of polypropylene is determined by the typical process when moulding. The X-ray diffraction pattern that shows the crystallinity of polypropylene on the composite is shown in Fig. 7.

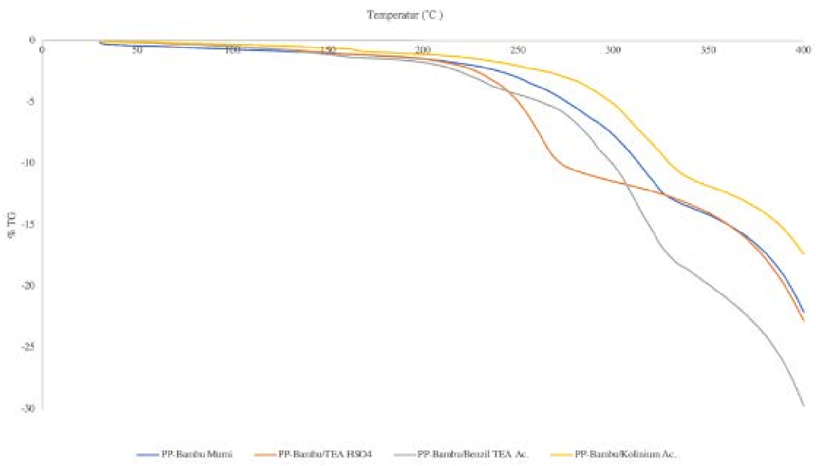

Fig. 6. Comparison of unmodified-pp and bamboo / IL-PP composite thermograms.

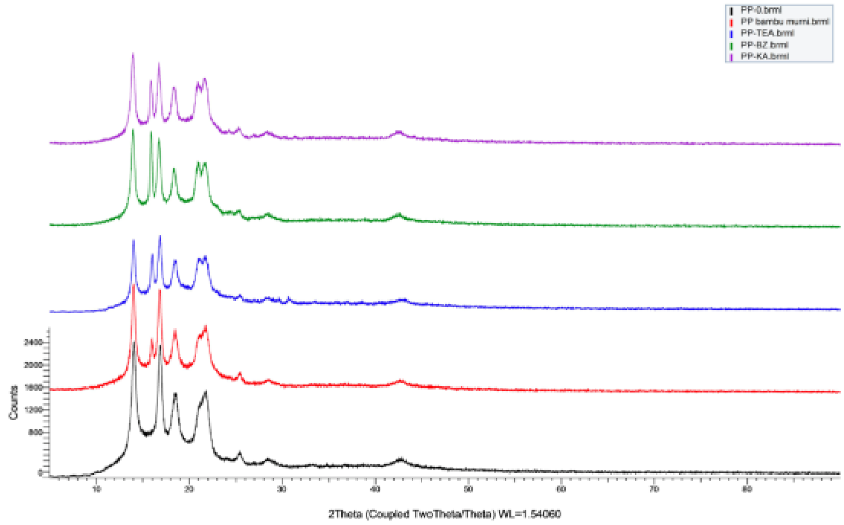

Fig. 7. Patterns of bamboo/IL-PP and x-ray bamboo unmodified-PP.

From the diffractogram, in Fig. 7 can be seen the emergence of a peak at 2θ = 16.2° which is the peak of the B-phase of polypropylene [1]. Based on comparative calculations of crystalline and amorphous peaks, the degree of crystallinity of composites with unmodified bamboo fillers and composites with bamboo fillers modified with ionic liquid triethylammonium hydrogen sulphate, benzyl-triethylammonium acetate, and calcium acetate are 47.70; 39.90; 49.30; and 48.6%, respectively.

The surface of the composite is observed using SEM to find out the surface of the composite. In Fig. 8, the surface of the composite shows the interaction between bamboo and polypropylene.

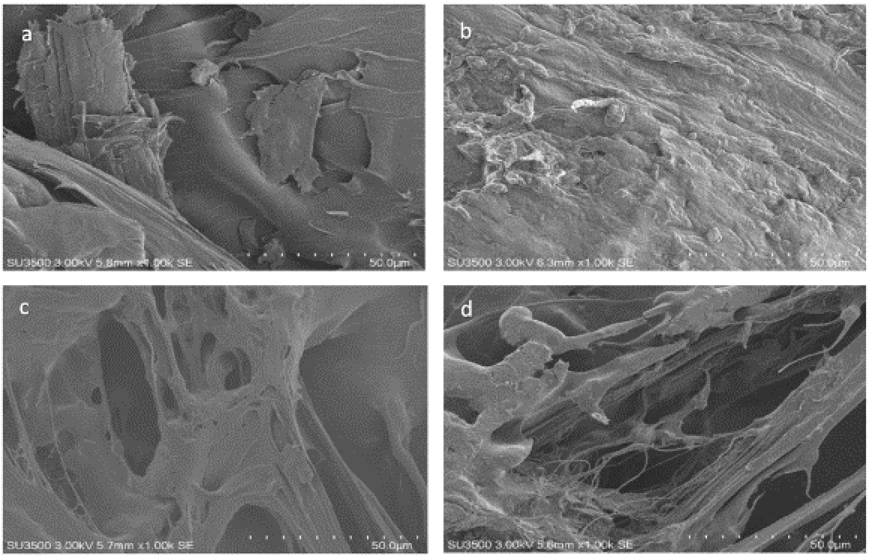

Fig. 8. SEM comparison of the appearance of unmodified-pp and bamboo/IL-PP: (a) pure bamboo (b) bamboo/[TEA] [HSO_4^-] (c) bamboo-benzyl [TEA] Ac. (d) bamboo-cholinium acetate.

Being based on Fig. 8, it can be seen that on composites with bamboo filler without modification (a) many bamboo faults do not interact with polypropylene. Whereas the composite with a modified bamboo triethylammonium hydrogen sulphate (b) composite surface is flat and the interaction between polypropylene and the bamboo may be good. In the composite with modified bamboo filler benzyl-triethylammonium acetate, the interaction of polypropylene with bamboo can be observed, and in the modified bamboo composition of aluminium acetate, the interaction between polypropylene and its bamboo is also seen.

In the outer layer of the untreated composite, Fig. 9(a), most bamboo flours are aligned in the direction of melt flow, and few bamboo flours are agglomerated. In the core layer, the bamboo flours are observed to randomly distributed in the matrix. In the composite treated with [TEA] [HSO_4^-], Benzyl [TEA] and Cholinium Acetate, the bamboo flours are randomly distributed in the matrix, and most are agglomerated, Fig. 9(b)-(d). A possible explanation can decrease the PP matrix content due to the incorporation of [TEA] [HSO_4^-], Benzyl [TEA] and Cholinium Acetate. Besides, compared to the untreated bamboo flours, the treated bamboo flours can be more hydrophilic due to the incorporation of the highly polar [TEA] [HSO_4^-], Benzyl [TEA] and Cholinium Acetate. The work of [20] shows similar results.

The mechanical test conducted is a tensile test to obtain tensile strength. Table 2 presents tensile strength and elongation at break values. It can be seen that the addition of bamboo or filler to the composite gives a higher tensile strength value compared to polypropylene without fillers. However, the elongation value of polypropylene is greater than that of the bamboo-polypropylene composite.

Tensile strength values between composites filled with bamboo filler without modification and bamboo resulting from ionic liquid modification increased tensile strength. In the modified bamboo, the ionic solution of cholinium acetate has the highest tensile strength value compared to the modification with triethylammonium hydrogen sulphate ionic liquid, and benzyl-triethylammonium acetate.

Table 2. Bamboo-PP composite mechanical test data

Sample	Tensile Strength (MPa)	Elongation (%)
PP	0.44	564.9
PP-Original Bamboo (Free O-H)	5.10	3.48
PP-Bamboo-$[TEA]^+$ $[HSO4]^-$	14.86	8.0
PP-Bamboo-[Benzil-TEA] $^+$ $[CH_3COO]^-$	14.31	3.9
PP-Bamboo-$[Cholinium]^+$ $[CH3COO]^-$	16.16	9.2

Values of all mechanical parameters in Table 2 obtained for PP are consistent with the literature [1]. Composites containing bamboo are characterized with slightly higher values of tensile strength and elastic modulus than the matrix. This strengthening effect observes for samples with ionic liquid treated bamboo may be attributed to better adhesion between components of the composite [18]. The increased adhesion, enabling more efficient stress transfer in the composite system, is most likely a result of the increased wettability of the bamboo fibres. This hypothesis is also supported by the research of Mahmood et al. [17], who investigated composites fabricated from lignocellulosic biomass pre-treated with ionic liquids.

4. Conclusions

Ionic liquids of triethylammonium hydrogen sulphate, benzyl-triethylammonium acetate, and calcium acetate are synthesized through quaternarization and anion changes. The structure of the ionic liquid of triethylammonium hydrogen sulphate, benzyl-triethylammonium acetate, and calcium acetate is confirmed by 1H-NMR and FTIR spectra. For thermal properties, ionic liquids are characterized using TG/DTA with decomposition temperature data of 265˚C triethylammonium hydrogen sulphate, 155˚C benzyl-triethylammonium acetate, and 196˚C collateral acetate. Modification of bamboo using ionic liquids causes changes in crystallinity, thermal stability, and tensile strength of bamboo-polypropylene composites. The crystallinity of the composite with unmodified bamboo fillers is 47.7%, with 39.9% modified triethylammonium hydrogen sulphate modified bamboo fillers, 49% modified benzyl-triethylammonium acetate bamboo filler, and 48.6% modified aluminium acetate bamboo filler. The temperature of the composite decomposition point with pure bamboo fillers is 234˚C, bamboo-triethylammonium hydrogen sulphate fillers 242˚C, bamboo-benzyl-triethylammonium acetate 267˚C fillers, and 276˚C bamboo-coletium acetate fillers, and 276˚C bamboo-cholinium acetate fillers. The tensile strength value of the bamboo composite filler without modification is 5.10 MPa, bamboo/triethylammonium hydrogen sulphate filler 14.86 MPa, bamboo/benzyl-triethylammonium acetate filler 14.3 MPa, and bamboo/cholinium acetate filler 16.16 MPa.

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