Journal of Material Sciences

# 100% Natural Biomass Thermoplastic Materials Incorporating Wood, Stone, and Natural Deep Eutectic Solvents Produced Using an Industrial Twin-Screw Extruder

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Research Article

**Received:** 28-May-2024, Manuscript No. JOMS-24-137493; **Editor assigned:** 31- May -2024, PreQC No. JOMS-24-137493 (PQ); **Reviewed:** 14-Jun-2024, QC No. JOMS-24-137493; **Revised:** 21-Jun-2024, Manuscript No. JOMS-24-137493 (R); **Published:** 28-Jun-2024, DOI: 10.4172/2321-6212.12.2.002

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**Citation:** Xiao S. 100% Natural Biomass Thermoplastic Materials Comprising Wood, Stone, and Natural Deep Eutectic Solvent Prepared by an Industrial Twin-Screw Extruder. J Mater Sci. 2024;12:006.





## ABSTRACT

Biomass-based renewable and biodegradable materials are attractive candidates for replacing non-biodegradable petrochemical-derived plastics. Herein, a wood-based thermoplastic material comprising wood, stone, and a Natural Deep Eutectic Solvent (NADES) was prepared by a facile method using an industrial twin-screw extruder. In addition, molding products were also obtained by simple injection molding. All of the components were derived from 100% biomass (assuming talc as biomass), with no materials originating from petroleum or fossil fuels. A deep eutectic solvent was used to dissolve a portion of the wood to afford a 100% biomass-based thermoplastic material by using a simple twin-screw extrusion process. The results revealed that the introduction of rosin or poly (3-hydroxybutyrate-*co*-3-hydroxyvalerate) led to the improvement in the mechanical strength and water resistance. Infrared spectroscopy (IR) and Nuclear Magnetic Resonance (NMR) spectral analyses revealed that cellulose, hemicellulose, and lignin were still present after treatment with NADES. As wood and stones are abundant and cost-effective materials, the as-prepared materials demonstrate novelty as new 100% bio-based thermoplastic materials.

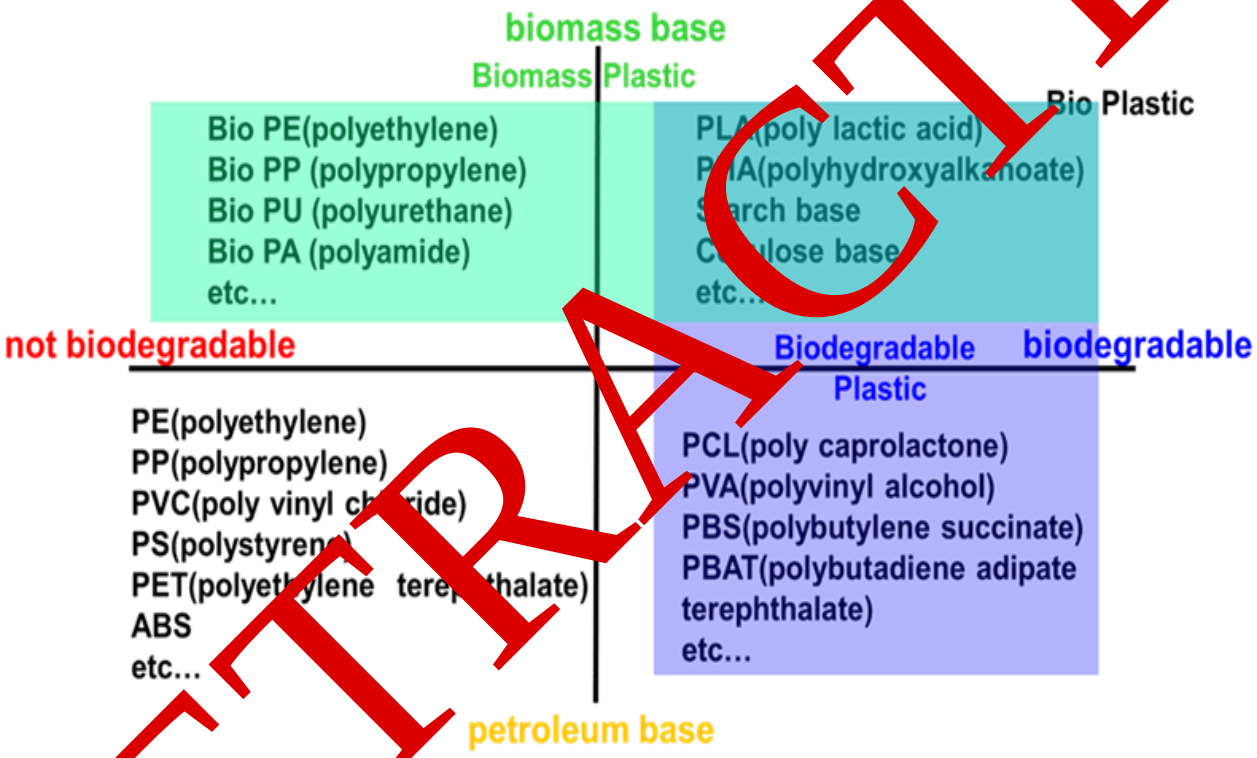
**Keywords:** Polyhydroxylalkanoate; Polylactic acid; Deep eutectic solvents; Flavonoids; Alkaloids.

## INTRODUCTION

Environmental issues such as climate change, global warming, natural resource depletion, and plastic pollution, etc., require urgent alleviate measures. Carbon dioxide ($CO_2$) is considered to be one of the causes of global warming. The atmospheric $CO_2$ content has been increasing intensively because of the burning and consumption of fossil fuels and petroleum by humans to produce chemical products and fuels to facilitate modern human lifestyle.

In this regard, plant-derived chemical products and fuels can be considered to be carbon-neutral because plants absorb $CO_2$ during their growth; hence, the total $CO_2$ emissions are less after the degradation of the used chemical products. Therefore, developing plant-biomass-derived chemical products is one approach to reducing $CO_2$ consumption for the purpose of suppressing global warming [1]. Plastics constitute a typical chemical product derived from petroleum. Preparing natural plant-biomass-derived plastics instead of petroleum-derived plastics is preferable. Moreover, ideally, biomass-derived plastics should exhibit biodegradability, which in turn can help in reducing the dreadful plastic pollution. Various bioplastics has been developed thus far. "Bioplastics" are categorized into "biomass plastic" and "biodegradable plastic." Depending on whether plastics are derived from biomass or petroleum and whether they exhibit biodegradability, all types of plastics can be categorized as shown in Figure 1 [2].

**Figure 1.** Classification of plastics according to the biomass and biodegradability.




Category 1 (right lower area) comprises petroleum-derived non-biodegradable plastics, which are neither produced nor consumed for over a century this category of plastics is a major contributor to plastic pollution. Polypropylene (PP, $[C_3H_6]_n$) and polyethylene (PE, $[C_2H_4]_n$) are representative examples of plastics. Category 2 (right lower area) comprises petroleum-derived biodegradable plastics. This category of plastics is derived from petroleum, but these plastics exhibit biodegradability. Typical examples include polybutylene succinate and Poly Butylene Adipate-*co*-Terephthalate (PBAT) [3]. Recently, these biodegradable plastics have been applied in agriculture, films, plastic bags, etc. Category 3 (left above area) comprises biomass-based non-biodegradable plastics, which are derived from plant-based biomass, but do not exhibit biodegradability. As a representative example, biomass PE has been used recently for shopping bags in Japan. This category of plastics can be regarded as carbon-neutral plastics because they do not emit $CO_2$ after their disposal as explained above [4]. According to this classification, ideal plastics are derived from biomass and are biodegradable. Category 4 (right upper area) can be regarded as these bioplastics, e.g., Poly Lactic Acid (PLA), starch-based biodegradable plastics, cellulose-based biodegradable plastics, and Poly Hydroxyl Alkanoate (PHA) [5-8]. Categories 2 and 4 comprise "biodegradable plastics," categories 3 and 4 comprise "biomass plastics,"

and categories 2, 3, and 4 are referred to as "bioplastics." By focusing on category 4 bioplastics in more detail, PLA and starch-based bioplastics are derived from food such as corn, sugarcane, etc. With the increase in food scarcity issues, PLA and starch based bioplastics are not ideal bioplastics.

In addition, rainforests can be destroyed by growing artificial palm trees, which are raw material sources for PHA. In this regard, cellulose-based bioplastics are the most ideal bioplastics as they can be obtained from non-edible biomass, and cellulose is an earth-abundant organic natural resource. However, obtaining cellulose-based bioplastics with high performance, such as high thermoplasticity and mechanical strength, is difficult using only cellulose-based materials. Therefore, petroleum-based additives are typically used to prepare cellulose-based bioplastics for real industrial applications [7]. If one cannot compromise to use any petroleum-based chemicals for plastics, then creating thermoplastic material from wood itself is ideal. Wood is the most desirable raw material for bioplastics because of their cost-effectiveness and environment-friendly nature. Even waste wood can be utilized as a raw material, which is a key advantage over the other types of bioplastics.

Meanwhile, Deep Eutectic Solvents (DESs), also known as low-melting mixtures, have been developed recently as a new breakthrough technology for the conversion of biomass into valuable chemicals [9]. DESs comprise a hydrogen-bond acceptor, typically a quaternary ammonium halide salt, coupled with a hydrogen-bond donor, such as an alcohol, an amino acid, an amine, a carboxylic acid, or a carbohydrate [10]. DESs, which are new, interesting materials, are ionic liquid analogs that are applied in several fields, such as metal electrodeposition, metal extraction, gas adsorption, electrolytes, organic synthesis, plasticizer, lubricants, extraction of plant-derived substances (flavonoids, alkaloids, etc.), and biotransformation, are under investigation [11]. In addition, some studies reported that wood-based resin analog materials can be prepared by the application of DESs for wood [12]. On the other hand, some wood-based polymer or composite materials were reported although they often contain petroleum-derived chemicals. In addition, wood is only applied as a filler in plastics [13]. However, to the best of our knowledge, wood-based thermoplastic materials with a 100% natural biomass composition have not been developed. Among various DESs, specific DESs referred to as Natural Deep Eutectic Solvents (NADES)s are reported. These are composed of only natural ingredients. Some NADESs can dissolve cellulose [14].

In this study, a Wood-And-Stone-Based Thermoplastic Material (WSTM) with 100% natural composition was prepared by using NADES. Furthermore, only a twin-screw extruder and an injection molding machine are essential to prepare WSTM; this approach exhibits a key advantage for real applications because these machines are already used in actual industries.

## MATERIALS & METHODS

Commercially available cherry tree (sakura) powder and talc (Imerys) were used as wood and stone raw materials, respectively. NADES was prepared by mixing lactic acid and glucose in a molar ratio of 2:1 at 120°C for 3 hrs. PP, lactic acid, and glucose were purchased from Wako Chemicals (Japan). Various types of WSTM with different compositions were prepared using a JSW twin-screw extruder model TEX30α (42:1 L/D ratio) at a screw rotation speed of 100 rpm. Its 12 heating zones were set at temperatures ranging between 180 and 210°C. Table 1 summarizes the prepared WSTM blend compositions. SEM images (JSM-6060LV, Jeol, Tokyo, Japan) were recorded at 1500× magnification to observe the surface morphology. Thermal analysis was performed using a TG–DTA system (Shimadzu, Japan). An Agilent Cary 630 Fourier Transform Infrared Spectroscopy (FTIR) spectrometer was employed to obtain the infrared spectra. Proton Nuclear Magnetic Resonance ($^1$H NMR) spectra were recorded using a Bruker Avance III HD 400 instrument at 400 MHz.

**Table 1A**. Composition of as-prepared WSTM with 100% natural composition without petroleum-derived chemicals.

| Wood | 30 | 40 | 50 | 50 | 50 | 50 | 50 | 50 | 50 | 50 |
|---|---|---|---|---|---|---|---|---|---|---|
| Lactic acid | 50 | | | | | | | | | |
| Glucose | 25 | | | | | | | | | |
| Talc | 50 | | | | | | | | | |
| Rosin | - | - | - | 25 | 50 | 75 | - | - | - | - |
| Phbv | - | - | - | - | - | - | 25 | 50 | 75 | - |
| Tensile strength ($N/mm^2$) | - | - | 3.1 | 9.8 | 11.2 | 13.9 | 7.2 | 10.6 | 13.1 | - |
| Bending strength ($N/mm^2$) | - | - | 2.3 | 13.8 | 14.9 | 16.1 | 8.9 | 9.1 | 11.2 | |
| Pelettization | x | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | x |
| Molding | x | x | 0 | 0 | 0 | 0 | 0 | 0 | 0 | x |
| Water resistance | x | x | x | 0 | 0 | 0 | 0 | 0 | 0 | x |

**Table 1B.** Composition of as-prepared WSTM combined with petroleum-based plastic (PE, PP).

| Wood | 50 | | | | | |
|---|---|---|---|---|---|---|
| Lactic acid | 50 | | | | | |
| Glucose | 25 | | | | | |
| Talc | 50 | | | | | |
| Pp | 25 | 50 | 75 | - | - | |
| Pe | - | - | - | 25 | 50 | 75 |
| Tensile strength ($N/mm^2$) | 13.8 | 15.7 | 7.5 | 8.1 | 9.6 | |
| Bending strength ($N/mm^2$) | 10.1 | 13.9 | 6.3 | 7.2 | 8.8 | |



## RESULTS AND DISCUSSION

First, WSTM was prepared using a twin-screw extruder as this extruder is an actual machine used for the industrial production of plastics. Table 1A summarizes the composition of the as-prepared WSTM. Talc was applied as stone. Notably, without talc, forming resin pellets with a twin-screw extruder was difficult. In addition, when the content of wood was less than a certain ratio, the resin pellets were not formed using the twin-screw extruder.

On the other hand, when the content of wood is greater than a certain ratio, the necessary force for pelletization becomes extremely strong such that wood-based pellets cannot be produced. Therefore, the contents of wood and talc need to be optimized to prepare WSTM pellets. NADES comprising lactic acid and glucose reportedly extracted botanical chemicals, such as anthraquinone, from plants [15].

On the other hand, lactic acid based NADES especially prepared with choline chloride is often examined for the dissolution of wood-based material [16]. In our study, chloride was not used for preparing NADES because hydrochloric acid may be formed during the twin-screw extruder process at high temperatures.

However, notably, even chloric-based chemicals were not used, the corrosion of the metal parts in the twin-screw extruder was observed. Therefore, metal parts should be coated with an anti-corrosion coating (e.g., fluoride-based coating), or Hastelloy should be used because of its corrosion resistance. Acidity possibly originating from lactic acid could be one of the reasons for possible corrosion.

Nevertheless, it was able to make test specimen for measuring mechanical strength when compositions were optimized by using an injection molding machine, which is a key advantage as injection molding is the most common

process for preparing plastic molding products among various molding processes, including blow molding, film and sheet molding, and vacuum molding.

However, water resistance is one of the drawbacks. Considering our goal to produce wood-based thermoplastic materials with 100% natural ingredients without petroleum-based chemicals, we selected only plant-biomass-based chemicals or bioplastics as additive materials, e.g., starch, PLA, poly (3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV), rosin, shellac, glue, and chitin. Starch was reported to be also weak in water [17].

PLA does not exhibit marine biodegradability. Shellac, glue, and chitin are not plant-based biomass; hence, rosin and PHBV are selected as natural additives to render water resistance. As a result, water resistance was rendered by the addition of rosin and PHBV. With an increase in the rosin or PHBV content, the mechanical strength of the plastics increased. Moreover, the improvement in the bending strength was more obvious upon the addition of rosin instead of PHBV [18].

Reported that when rosin was added into a PLA/PBAT composite, the mechanical strength was improved because of compatibility enhancement and plasticizer effects. In addition, WSTM was mixed with conventional petroleum-based plastics such as PE and PP (Table I (b)). WSTM exhibited good compatibility with these petroleum-based plastics, and its mechanical strength and water resistance were improved. Although these plastics still contain petroleum-based chemicals, they contribute to partially reducing $CO_2$ emissions and plastic pollution by using wood and stone as the raw materials.

Hence, WSTM can be used as a bulky agent for the plastic industry because of its cost-effectiveness. However, water resistance is one of the drawbacks. Considering our goal to produce wood-based thermoplastic materials with 100% natural ingredients without petroleum-based chemicals, we selected only plant-biomass-based chemicals or bioplastics as additive materials, e.g., starch, PLA, poly(3-hydroxybutyrate-*co*-3-hydroxyvalerate) (PHBV), rosin, shellac, glue, and chitin. Starch was reported to be also weak in water [17]. PLA does not exhibit marine biodegradability. Shellac, glue, and chitin are not plant-based biomass; hence, rosin and PHBV are selected as natural additives to render water resistance. As a result, water resistance was rendered by the addition of rosin and PHBV. With an increase in the rosin or PHBV content, the mechanical strength of the plastics increased.

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Figure 2 shows the SEM image of the as-prepared WSTM. Parts of wood and stone (talc) were observed. Even NADES was reported to dissolve wood; hence, unreacted or undissolved wood was observed. The percentage of dissolved cellulose, lignin, or wood critically depends on the DES composition and percentage with respect to the wood weight as well as the reaction temperature [12]. The dispersibility of wood and stone can be improved by further mixing using a twin extruder although excessive blending may deteriorate the quality of the WSTM; hence, mixing conditions should be optimized.

**Figure 2.** Top-view SEM images of as-prepared WSTM. The weight composition ratio of this WSTM is 50:25:50:40 (lactic acid:glucose:talc:wood).

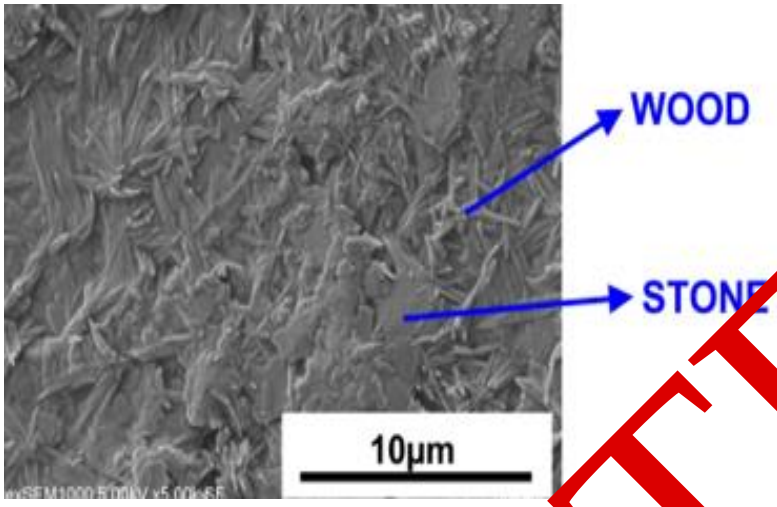



Figure 3 shows the TG–DTA results of as-prepared WSTM. The majority of the lactic acid and glucose (NADES composition) should be decomposed at temperatures of less than 300 °C, which was not observed in our experiment [19, 20]. A small peak at 368 °C in the DTA curve exhibited a relatively good agreement with the DTA peak assignment in a wood thermal decomposition study reported previously. This is despite that we observed a more obvious peak at 423 °C-445 °C that corresponds to a slightly higher decomposition temperature than that reported in previous wood decomposition studies [21]. The reason for the increase in the decomposition temperature of WSTM is not clear at this stage. It could be suggested that the physical properties of compressed and solvated wood changed during WSTM preparation using a twin-screw extruder at high temperatures. At 700 °C, the remaining weight ratio was approximately 33%, corresponding to the weight of talc in the WSTM.

**Figure 3.** TG-DTA curves of as-prepared WSTM. The weight composition ratio of WSTM is 50:25:50:40 (lactic acid/glucose/talc/wood ratio).

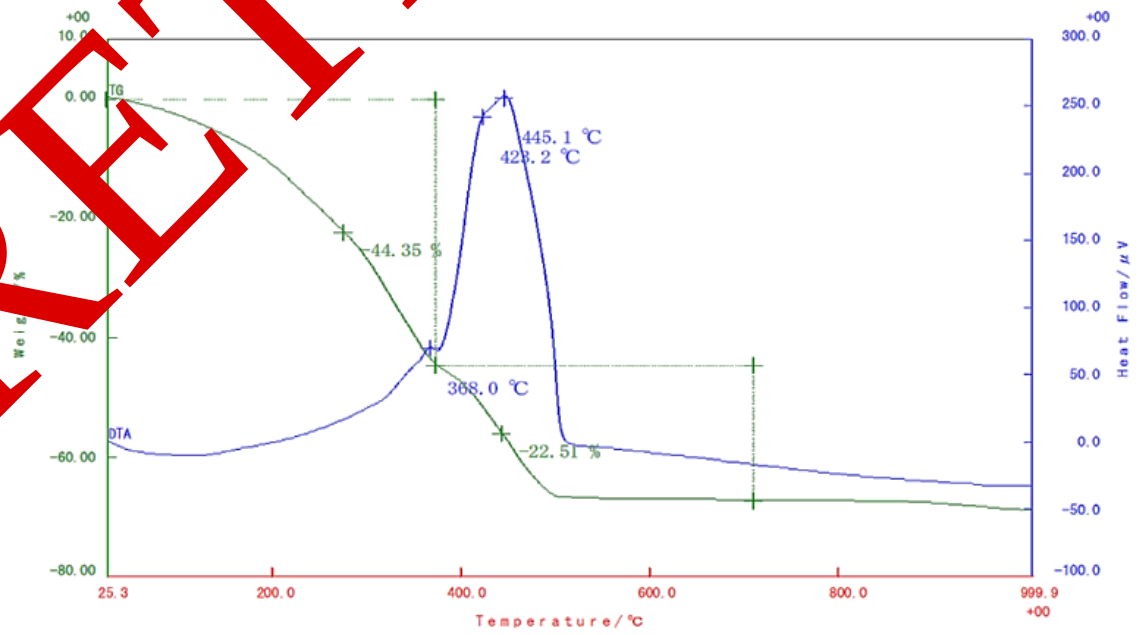


Figure 4 shows the IR spectra of WSTM. A wide absorption at 900-1200 $cm^{-1}$ corresponded to the O-H group in the glucose structure, and an absorption corresponding to the $H_2O$ of cellulose was observed at 1650 $cm^{-1}$. The peak at 1034 $cm^{-1}$ corresponded to the C-O stretching vibration of cellulose ether bonds. Furthermore, the C-O stretching vibrations of lignin and hemicellulose were observed at 1243 $cm^{-1}$, whereas the aromatic nucleus skeleton vibration was observed at 1505 $cm^{-1}$.

In addition, the C-H stretching vibration and O-H stretching vibration were observed at 2925 and 3375 $cm^{-1}$, respectively [12]. From these observations, the high-temperature twin-screw extruder process promoted wood dissolution; hence, these IR absorption peaks characteristic of cellulose and lignin were observed. Wang et al. also claimed that DESs dissolved lignin *via* the cleavage of a lignin–carbohydrate complex and β-O-4 linkages [13]. Furthermore, also reported that DESs increased the hydrolysis efficiency of cellulose [23]. Therefore, wood was speculated to be dissolved to show cellulose and lignin in our preparation procedure.

**Figure 4.** IR spectra of as-prepared WSTM. The weight composition ratio of WSTM is 50:25:50:40 (lactic acid:glucose:talc:wood).

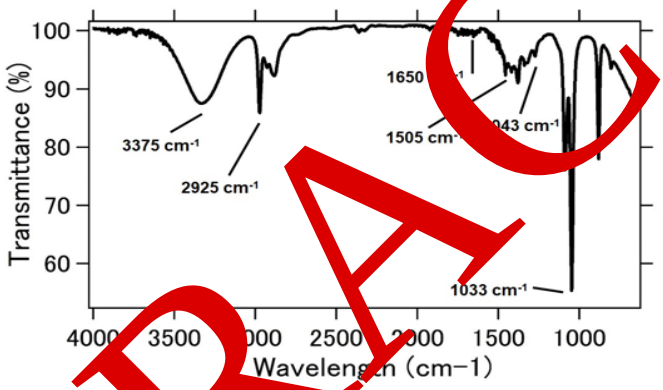
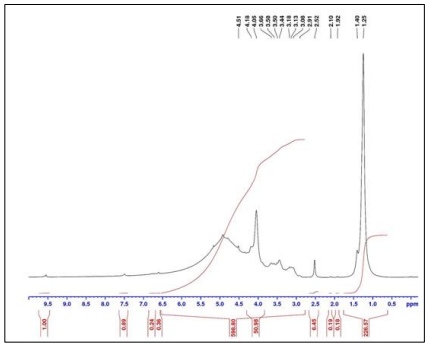



Figure 5 shows the $^1H$ NMR spectrum of WSTM. The peak at 1.2-1.4 ppm corresponded to the protons in aliphatic and aromatic acetates. The methyl group peak should be observed at approximately 1.8 ppm although a clear peak was not observed. These peaks were thought to be derived from lignin. In addition, the signal observed at approximately 2.4-2.5 ppm corresponded to protons from aliphatic acetates and aromatics or the acetyl group of hemicellulose [24]. The peaks observed at approximately 3.5 and 4.0 ppm were thought to be the Hγ of β-O-4, Hβ, or the methyl protons of 4-O-methyl-α-D-glucuronic acid [25]. The peak at approximately 5.0 ppm could be assigned to Hα of β-O-4 [26]. All of these peaks confirmed the presence of cellulose, lignin, and hemicellulose, which were consistent with the IR spectral data.

**Figure 5.** $^1H$ NMR spectra of as-prepared WSTM. The weight composition ratio of WSTM is 50:25:50:40 (lactic acid: glucose:talc:wood).



Reported that DES is effective in removing some of lignin and hemicellulose from wood, while the C-C bonds in lignin were unaffected, allowing most of the characteristics and activities of natural lignin to be maintained [27]. In contrast to traditional methods such as strong acid or alkali treatment, DES treatment is a physical dissolution process rather than a chemical decomposition process such that DES delignification treatment does not damage the cellulose structure, and the stiff cell wall becomes more flexible. Therefore, this physical wood dissolution process by DES thought to render thermoplastic characteristics, which was effective in the twin-screw extruder process for obtaining pellet like morphology.

## CONCLUSION

Wood- and stone based thermoplastic materials without any petroleum derived chemicals were successfully prepared by introducing wood, talc, and NADES in a twin-screw extruder mixing. In addition, it was possible to produce a molding product just by simple injection molding. The water resistance of WSTM was improved by adding rosin and PHBV, and mechanical strength was enhanced, while a 100% natural composition was maintained. WSTM also exhibited good compatibility with petroleum-based plastics such as PE and PP, which also enhanced its mechanical strength and water resistance. The IR spectra and NMR results suggested that NADES enhanced wood dissolution under high-temperature treatment using the twin-screw extruder. Besides wood, other organic waste such as waste wood, waste paper, and waste seaweed could also be used so long as they contain cellulose and lignin-based materials.

## AUTHOR CONTRIBUTIONS

Ryohei Mori drafted original draft, methodology, data curation, visualization, review, editing, supervision and conceptualization.

## CONFLICTS OF INTEREST

There are no conflicts to declare.

## ACKNOWLEDGEMENTS

The author wishes to thank Dr. Hideki Yoshioka, Mr. Daiki Tanaka, Mr. Yoshiyuki Konishi, Mr. Kenji Tomatsu, Mr. Yoshiteru Sato for their helpful discussions.

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