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Development of UV 3D-Printing Resin Using Polyhydroxybutyrate and Low-Methoxyl Pectin From Durian Rind

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Low Methoxyl Pectin (LMP), UV 3D printing, Polyhydroxybutyrate (PHB), Durian Rind

Abstract

Durian rind is often discarded as agricultural waste, contributing to environmental issues. Furthermore, the use of biodegradable materials in 3D printing may lead to sustainable and recyclable items. Pectin's high gel-forming ability, biodegradability, and biocompatibility make it an ideal biopolymer. The purpose of this study is to extract and characterize low-methoxyl pectin (LMP) from durian rind for potential use in UV 3D printing. The methoxyl content of pectin extracted from durian rind is 1.37 ± 0.601%. The resulting resin is then exposed to UV light and analyzed using FTIR to confirm polymer formation and functional group changes. The resin commercial exhibits minor changes in functional groups, such as a slight decrease in the intensity of O-H and N-H stretching peaks 3440 to 3507 cm^{-1} change 3437 cm^{-1} range after exposure to UV light and a change in the C-O 1271–1059 cm^{-1} range stretching band from 1256 to 1062 cm^{-1} after UV exposure comparing the resin formulation remains had same peak even after UV exposure, with no significant changes in its functional groups.

1. Introduction

In recent years, 3D printing has gained widespread popularity across various industries, including agriculture, food production, healthcare, automotive, and aerospace. This innovative technology is revolutionizing manufacturing by offering sustainable, cost-effective, and customizable solutions [1]. One of the most promising aspects of 3D printing is its potential to reduce waste, particularly in the context of food and agricultural byproducts. By using recyclable materials and allowing for the on-demand production of goods, 3D printing offers a sustainable approach to manufacturing. Specifically, photopolymerization the process of curing photo-reactive polymers using ultraviolet (UV) light has emerged as the primary method in 3D printing, especially in the creation of complex, custom-designed objects [2].

The need for biodegradable and environmentally friendly materials in 3D printing has led to significant interest in plant-based polymers that can reduce waste and encourage recycling. Agricultural byproducts, such as durian rind, are often discarded, contributing to environmental pollution. However, durian rind contains valuable compounds, such as low-methoxyl pectin (LMP), which could be utilized to develop sustainable materials for 3D printing. In regions like Vietnam and other ASEAN countries, where large quantities of durian rind are produced annually, turning this waste into valuable resources could play a critical role in advancing a circular economy. For instance, Vietnam produces up to 80,000 tons of durian fruit each year, much of which ends up as waste in landfills [3]. This agricultural waste contains potentially valuable compounds that can be

harnessed for sustainable applications [4]. This approach not only supports sustainable waste management but also promotes the use of renewable materials in innovative applications [5]

One such application is the development of resin formulations for UV-based 3D printing. In this study, we focus on extracting and characterizing LMP from durian rind to explore its potential as a key component in resin formulations for 3D printing. The use of LMP, in combination with polyhydroxybutyrate (PHB), offers a biodegradable and compostable alternative to traditional petroleum-based polymers, aligning with the principles of the circular economy [6]. Additionally, the curing process of the resin under UV light will be analyzed using Fourier Transform Infrared (FTIR) spectroscopy to confirm polymer formation and monitor changes in functional groups [7].

The research aims to extract and characterize low-methoxyl pectin (LMP) from durian rind for potential use in resin. Furthermore, this study is to analyse resin curing under UV light using FTIR to confirm polymer formation and functional group changes.

2. Methodology

2.1 Material

Durian rinds were bought from a small stand at Panchor, Johor. All the chemicals used were of analytical grade. The following material were utilized in this study are Polyhydroxybutyrate (PHB) ,1 N hydrochloric acid, 95% ethanol, Chloroform, Acrylate, Glycerol, Acetone, sodium chloride, phenol red indicator, 1 N sodium hydroxide, calcium chloride, Laccase, Isopropyl Alcohol, Citric Acid, Sodium Phosphate Monobasic was used in this study.

2.2 Equipment and Instruments

The following instrument that will be used was hot air oven (Memmert D06836,Germany), a bench-top centrifuge (Kubota J100, Fujioka, Japan), 3D printing Machine (Any Cubic Photon Mono, China), Grinder (Micro Universal Bench Top Grinder, Retsh ZM 100, Germany), Fourier transform infrared Spectroscopy (FTIR) (Shimadzu IRAffinity-1S, Japan), pH meter, Oven Chamber, Refrigerator and Desiccator.

2.3 Method

2.3.1 Preparation and Extraction of Low-Methoxyl Pectin from Durian Rinds

One of the most important steps in the extraction process is choosing an appropriate solvent for phytochemical extraction [8]. The durian rinds have been removed of their rough and rough edges. Among the extracting solvents, acid extraction is advantageous because it produces a higher yield of pectin and the extracted pectin is generally enriched in galacturonic acid, reflecting a greater purity [23]. The durian rind's remaining white and creamy portion was chopped into uniformly sized, tiny chunks that were roughly 1.5 cm. The durian rinds were then dried individually in a hot air oven according to variety on metal trays at 60 °C for 24 hours [9]. When extracting pectin, using a higher temperature can cause damage to the molecules that form the pectin chain, resulting in a decrease in yield and a reduction in pectin quality. This is because the high temperature can cause the pectin to crack or degrade, leading to a lower molecular weight and viscosity of the pectin [23]. Pectin extraction from dried durian rind powder was done by a modified method. 900 mL of HCL (1 M) was progressively combined with 100 g of powdered dry durian rind until the pH reached 2.5. Low pH may promote quick disruption of hydrogen bonds and ester linkages between pectin and cell wall, which increase the rate of diffusion of pectin and pectin extraction [23]. After that, it was incubated in a water bath at 85°C for four hours. The solution was then filtered through cheesecloth to produce the filtrate, which was then allowed to settle in the open air. After that, the filtrate was combined with acidified ethanol (4% HCL + 95% ethanol, 1:4) and centrifuged for 20 minutes at 4000 rpm. Two more washes with 95% ethanol were performed on the solution. After that, the extracted pectin in the solution's precipitate were gathered and dried for 24 hours at 55°C until they reached a constant weight. The manufacture of biopolymers made use of these dried extracted pectin [7]

2.3.2 Preparation of Dilution Polyhydroxybutyrate (PHB)

To prepare the Polyhydroxybutyrate (PHB) solution, chloroform was used as a solvent to dilute the PHB. Weight 1 gram of Polyhydroxybutyrate (PHB) and 100ml of Chloroform was measured. After that, added Polyhydroxybutyrate (PHB) and Chloroform into a beaker. The mixture was stirred by using magnetic stir until it was clear and homogenous [10]. The concentration of PHB is 10 mg/mL when 1 gram is added to 100 millilitres of chloroform.

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2.3.3 Preparation of pH Buffer (5.5)

To prepare a pH buffer of 5.5, sodium phosphate monobasic (NaH_2PO_4) were used. First, 0.1 M sodium phosphate monobasic stock solutions were made by weighing 6.9 grams using an accurately weighed and dissolving them with 100 millilitres of purified water. Using a pH meter, the solution's pH was determined, and NaOH was gradually added until it reached 5.5. Finally, the solution was diluted using distilled water until the total volume reached 900ml. The prepared solution was stored in a labelled container.

2.3.4 Preparation of Calcium Chloride ($CaCl_2$)

To prepare the calcium chloride ($CaCl_2$) solution, 10.95 g of calcium chloride was weighed using an accurately weighed. The measured calcium chloride was transferred into a beaker, and a small amount of distilled water was added. The mixture was stirred continuously until the calcium chloride was completely dissolved. After ensuring complete dissolution, distilled water was added into the solution until the total volume reached 500ml. The prepared solution was then stored in a labelled container.

2.3.5 Preparation to mixer all ingredients (Resin Formulation)

The preparation of the mixture involved combining low-methoxy pectin, PHB, pH buffer (5.5), acetone, glycerol, calcium chloride ($CaCl_2$), acrylate, isopropyl alcohol, and laccase ingredients. Next, the pH buffer (5.5) was prepared and stored in refrigerator. Low-methoxy pectin and PHB were dissolved separately in beaker as for chloroform for PHB and pH buffer 5.5 for pectin using magnetic stirring to ensure complete dissolved. The mixture was gradually added with acetone, glycerol, and isopropyl alcohol to ensure uniformity. The addition of acetone, glycerol, and isopropyl alcohol helped to modify the viscosity and consistency of the mixture, optimizing it for further processing. Calcium chloride ($CaCl_2$) and acrylate were then poured into the solution to enhance cross-linking and polymerization properties. Finally, laccase was added as a catalyst to facilitate the desired chemical reactions. The solution was stirred continuously using a magnetic stirrer to ensure homogeneous and complete dissolved of all ingredients, resulting in a resin formulation suitable for further processing in UV 3D printing [11].

2.3.6 Characterization of Low-Methoxyl Pectin by Titration Method

Titration techniques were used to characterize pectin according to its methoxy concentration. 250 ml of conical flask was filled with 0.5 g of durian rind pectin and 5 ml of ethanol. Next, 100 ml of distilled water was added and 1g sodium chloride was added into the solution. After that drop six of phenol red to the solution as an indicator, it will be titrated against 0.1 N sodium hydroxide. The solution will be in purple or pink titration points. The neutral solution will be combined with 25 ml of 0.25 N sodium hydroxide and stirred it until it homogenous. The solution was left for 30 minutes at room temperature. After that, 25 ml of 0.25 N hydrochloric acid was used into the solution and titrated against 0.1 N sodium hydroxide. The methoxy content of pectin was determined and calculated as below [12].

$$\text{Methoxyl content (\%)} = \frac{\text{mL of Alkali} \times \text{Normality of alkali} \times 3.1}{\text{eight of sample}}$$

2.3.7 Fourier Transform Infrared Spectroscopy (FTIR) Method

A Fourier Transform Infrared Spectroscopy (FTIR) spectrometer (Shimadzu IRAffinity-1S, Japan) will be used to analyse the spectra of the resin before and after UV curing, helping to identify changes in the chemical structure of the material. The sample of the film was cut into a small square, or a few drops of liquid resin were placed in the sample holder. The sample was positioned below the ATR crystal of the FTIR spectrometer. The spectrometer was turned on, and the spectrum resolution was set at 4 cm^{-1} with 64 spectra collected Between 400 and 4000 cm^{-1}, the wavelength range was modified. The sample's infrared spectrum data was gathered to analyze the peaks and determine the features. The chemical bonds and functional groups present in the formulation were determined. The peaks were compared between the spectra obtained before and after curing to observe changes in the chemical structure. [13].

3. Result and Discussion

3.1 Methoxyl Content of durian rind pectin

In this study, the methoxy content of pectin obtained from durian rind was determined by titration method. According to result from titration, it is found that the methoxyl content of pectin extracted from durian rind is

1.37 ± 0.601%. If methoxyl content was less than 7%, then it would be classified as low in methoxyl [14]. The high methoxyl pectin was contain greater than 7%, to making them high-methoxy pectin [12] The methoxyl content values in this study indicated that pectin from durian rind was lower than 7%. Thus, it was categorized as low methoxyl pectin (LMP). These low methoxyl pectin will be able to form a gel in the presence of polyvalent cations such as calcium [14].

3.2 Fourier Transform Infrared Spectroscopy (FTIR)

Fig. 1 shows that at the low-methoxyl pectin results. To verify the and determine the primary features of functional groups of the pectin, further FTIR analysis of the extracted pectin was carried out. This broad peak contain has high absorbance in region 3284 cm^{-1} is typically contain O=H and C-H stretching vibration, mostly from functional group in range of class, Alkynes, Carboxylic acids, Alcohol and Hydroxyl compound. The O-H stretching absorption caused by between and intermolecular hydrogen bonding of the galacturonic acid polymer in pectin was represented by a strong and broad absorption region between 3600 cm^{-1} and 3200 cm^{-1} [9]. the spectra revealed peaks at 3340 cm^{-1} and 3441 cm^{-1}, indicating the presence of -OH groups, the predominant functional groups in cellulose. The intensity of these peaks deepened after acid hydrolysis and pretreatment, suggesting an increase in the hydrophilic characteristics of cellulose, primarily due to a higher concentration of hydroxyl groups in the fibers [22]. The peak at 1636 cm^{-1} is from C=C that particularly contain alkenes compound that has medium intensity. Next, the region 2400 to 1900 cm^{-1} range is not represented, as it is of little informative value [15]. According to [16] the FTIR testing of durian rind samples contained O-H (Hydroxyl group), C-H (Hydrocarbon or Alkanes), C≡C (Alkyne), C=O (Carboxylic group) and C=C (Alkene) functional groups.

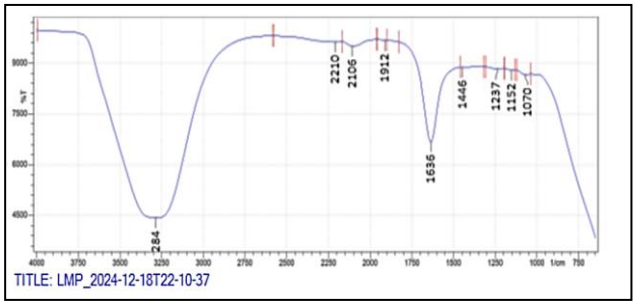

Fig. 1 FTIR spectra of Low-methoxyl pectin (LMP)

Fig. 2 shows the Polyhydroxybutyrate results. To verify and identify the main characteristics of specific functional groups in the ingredient, further FTIR analysis of the polyhydroxybutyrate was carried out. The sharp absorption band at 742 cm−1 which corresponds to C-C stretching vibration, having strong intensity and mostly from functional group in range of class Alkyl halides compound. The peak at the 1211 cm−1 shows strong intensity that polyhydroxybutyrate had C-O that usually in functional group of Esters compound. According to [16] these peaks denote carbonyl C=O and asymmetric C-O-C stretching vibration, respectively, characteristic for ester bonding found in PHB molecule. The infrared spectra of all samples have a broad band between 1000 cm-1 and 1300 cm-1 range. Since these unsaturated bonds are essential to the photopolymerization process, which enables the material to cure and solidify when exposed to UV light, this is especially pertinent to UV 3D printing. All things considered, PHB's ester bonds and alkenes make it an appropriate choice for UV 3D printing because these functional groups allow the material to crosslink and solidify when exposed to UV light, which enhances its printability and suitability for solid structure formation. This band consists of a strong peak [8]. The C-O and C-C vibrations of stretching were caused by the absorption bands at 1138 cm-1 to 829 cm-1 range, which unstructured PHB could achieve [17]. The peak at band position 3019 cm−1 consists functional group of Alkenes C-H that has medium intensity. Polyhydroxybutyrate usually had four essential elements C, H, O, and N are required for cells to function physiologically normally. PHB is highly crystalline due to its linear chain structure, containing both amorphous and crystalline phases. It can be found as a virgin polymer or as part of copolymers and blends. It is generated as a carbon reserve in a wide variety of producing bacterial strains and is produced industrially through bacterial fermentation. PHB also has several advantages over synthetic polymers to produce certain packaging applications. The main source of H and O is water. However, depending on how they eat, bacteria get their C and N from various sources [17].

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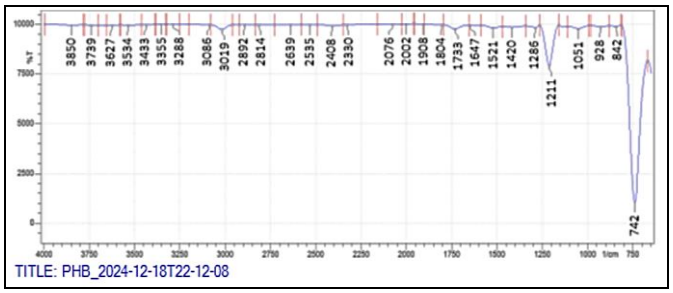

Fig. 2 FTIR spectra of Polyhydroxybutyrate (PHB)

Fig. 3 shows the resin formulation results. To confirm and identify the main characteristics of functional groups of the component, further FTIR analysis of the resin formulation was carried out. This broad peak contain has high absorbance in region 3321 cm^{-1} is typically contain N=H, O=H and C-H stretching vibration, mostly from functional group in range of class, Alkynes, Amines and Alcohol compound. Moreover, O=H is Functional group that usually the peak appears much broader than the other IR absorption. The O-H stretching absorption caused by between and intermolecular hydrogen bonding of the galacturonic acid polymer in pectin [9]. The peak at 1636 cm^{-1} is from C=C that particularly contain alkenes compound that has medium intensity. Next, the region 2400 to 1900 cm^{-1} range is not represented, as it is of little informative value [19].

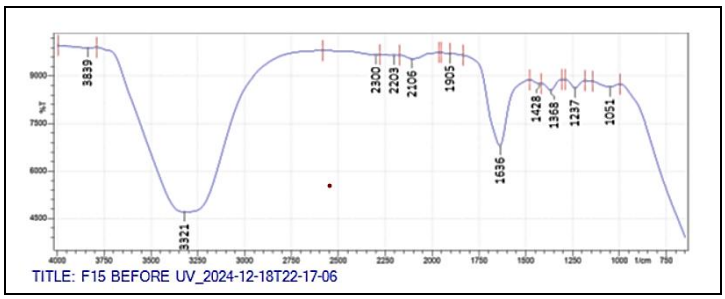

Fig. 3 FTIR spectra of resin formulation that contain all ingredients (of low-methoxyl pectin, PHB, pH buffer 5.5, Acetone, Glycerol, $CaCl_2$, acylate, Isopropyl Alcohol and Laccase)

Fig. 4 shows the formulation 15 before and after UV exposure. The absence of changing in peak or functional group indicates that the sample remains stable and not affected by UV exposure. As the result the O-H stretching region at 3321 cm^{-1} have remained broad and having no degradation of hydroxyl group. The peak at 1636 cm^{-1} and other at 1428 cm^{-1} to 1051 cm^{-1} range have remain stable indicates the chemical bonds like Alkynes, Amines and alcohol are unaffected by UV exposure. So that it shows the ingredients that was used in resin formulation likely does not undergo Photopolymerization such as bond breaking or oxidation under UV exposure. If the resin does not undergo the necessary functional group changes or crosslinking when exposed to UV light, it would not solidify as required for 3D printing. In this case, the resin would not be able to cure or harden properly, making it ineffective for the intended application. Therefore, while stability is beneficial for long-term material durability, it may also prevent the resin from performing as required for UV 3D printing.

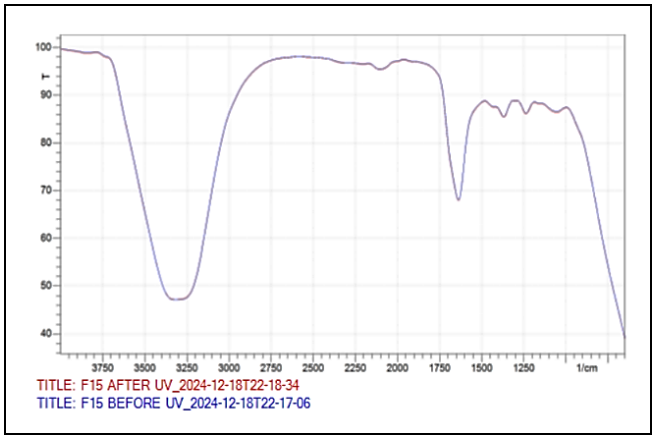

Fig. 4 Overlay FTIR spectra of resin formulation that contain all ingredients (of low-methoxy pectin, PHB, pH buffer 5.5, Acetone, Glycerol, $CaCl_2$, acylate, Isopropyl Alcohol and Laccase) after and before UV light exposure

Fig. 5 and Fig. 6 show the FTIR spectra of resin commercial before and after UV light exposure. FTIR analysis was conducted in resin commercial was conducted to confirm and identify the main characteristics of specific functional groups of the ingredient. The peak at band position 3440 cm^{-1} to 3507 cm^{-1} range show that resin commercial has O=H and N=H stretching absorption that have strong and broad intensity, the compound mostly from functional group in range of class, Amines, Alcohol and Hydroxyl. The changes in the functional groups are generally significant. The decrease in the O-H and N-H stretching peaks indicates that the resin is undergoing successful curing and polymerization under UV light. This peak is commonly associated with hydroxyl groups O-H present in alcohols, phenols, or water molecules. The decrease in the intensity of this peak suggests a reduction in the amount of free hydroxyl groups or water content. The N-H stretching band is typically associated with amines or amides. A decrease in this peak may indicate a reduction in the available amine groups, likely due to their involvement in the polymerization process, such as in reactions with acrylate or other reactive components in the resin formulation. While the peak after UV exposure has decrease into 3437 cm^{-1} indicate the chemical group like Amines, Alcohol and Hydroxyl are affected by UV exposure. The small peak at band position 2956 cm^{-1} to 2881 cm^{-1} range had C-H that had medium to strong intensity with Alkanes, alkyl group of functional class. Before exposure to UV light, the peak in resin commercial was slightly lower than after, and the number of absorption bands changed, but the functional group remained unaffected. In resin commercial the absorption band in 1718 cm^{-1} range, the bond C=C stretching and had weak intensity in Alkene compound. For example, the mixture of cycloalkanones show characteristic differences of the carbonyl group cyclobutanone 1788 cm^{-1}, cyclopentanone 1746 cm^{-1} and cyclohexanone 1718 cm^{-1} [18]. The alkene compound has unaffected during UV exposure. The functional group found in the absorption band in 1401 cm^{-1} range, the bond N=O bend from Nitro group compound. From absorption band in 1271 to 1059 cm^{-1} range had C-O stretching and had strong intensity, had Esters compound. The peak was slightly lower before exposure to UV radiation than it was after, and the number of absorption bands changed, but the functional group remained unaffected. The small increase in the region from 1059 to 1062 cm^{-1} is probably the result of small bonding or change in structure imposed on by UV radiation, such as weaker hydrogen bonds or minute chemical rearrangements. The sensitivity to UV-induced interactions is shown by the change, which is usually not indicate of major degradation. Next, the region 600 - 900 cm^{-1} range is not represented, as it is of little informative value [19].

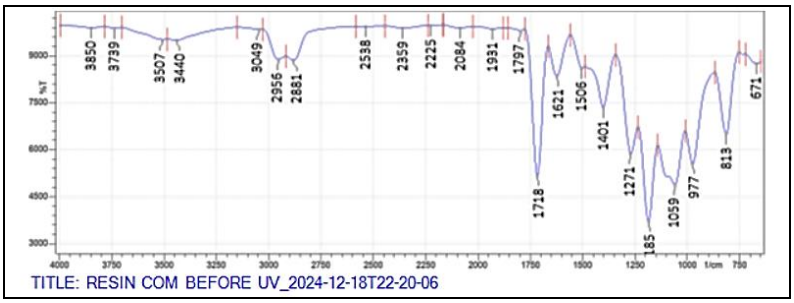

Fig. 5 FTIR spectra of Resin Commercial Before uv light exposure

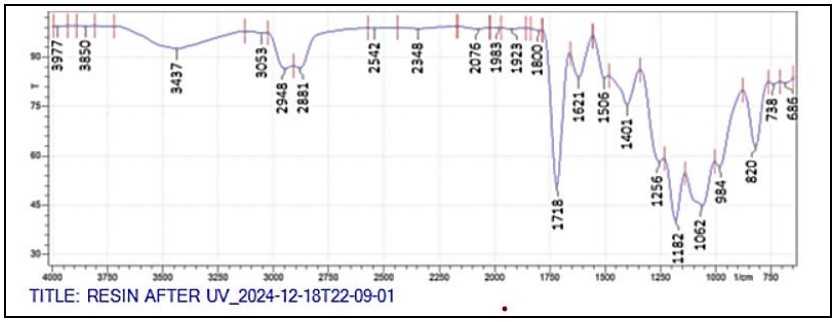

Fig. 6 FTIR spectra of Resin Commercial After uv light exposure

The difference between the before and after UV exposure has shown in Fig. 7. The functional groups in commercial resin had small changes because of UV-induced interactions, according to the FTIR analysis completed before and after UV exposure. Following UV irradiation, the broad peak in the 3440–3507 cm^{-1} range that was given to O–H and N–H stretching from amines, alcohols, and hydroxyl groups somewhat reduced showing that these groups were affected. Like this, after being exposed to UV light, the peaks in the 2956–2881 cm^{-1} range which represent the C–H stretching of alkanes and alkyl groups somewhat increased in intensity, indicating either improved bonding interactions or structural change. UV irradiation had no effect on the nitro

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group bond at 1401 cm^{-1} or the absorption band at 1718 cm^{-1}, C=C stretching in alkenes, showing the same in these.

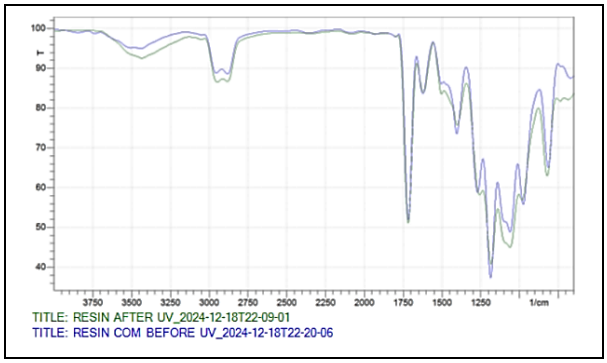

Fig. 7 FTIR spectra of Resin Commercial of after and before UV light exposure

Fig. 8 shows the resin formulation had the comparison in the absorption peak, functional group and type of vibration in between the resin commercial after UV exposure. The resin used in UV 3D printing needs to go through a process known photopolymerization, that produces solid layers by curing and hardening the material when exposed to UV light. Functional groups such as O-H, N-H, and C-H stretching bands are not affected when exposed to UV light, which suggests that the resin does not go through the required crosslinking reactions. This indicates the resin is unsuitable for this use since it is not photopolymerizable and would not harden as needed for 3D printing. Some functional groups in the resin commercial exhibit slight changes during UV exposure, especially those linked to alcohols, amines, and hydroxyls, showing a minor impact. However, there is not an evident change in these functional groups in the resin formulation. The resin formulation was unaffected or had chemical change during the UV exposure. This shows that the ingredients that was choice to the resin formulation had unaffected during the UV exposure while the resin commercial having the slightly change in the absorbance band. While the resin's stability may ensure its durability in other applications, its lack of ability to go through photopolymerization makes it unsuitable for UV-based 3D printing.

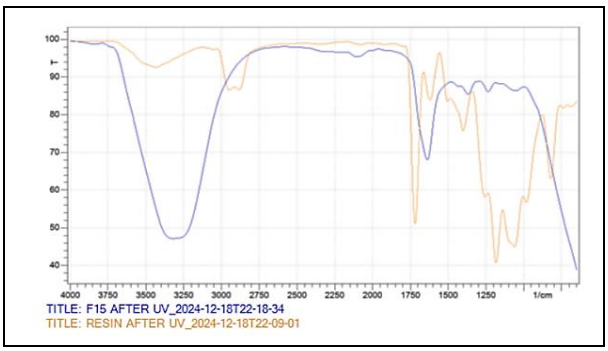

Fig. 8 FTIR spectra of resin commercial and resin formulation in this study of after UV light exposure

4. Conclusion

In this study the methoxyl content of pectin extracted from durian rind was categorized as low methoxy pectin. This shows the potential of resin formulations containing low-methoxy pectin (LMP) for UV 3D printing through Fourier Transform Infrared Spectroscopy (FTIR). Low methoxy pectin can form gels in the presence of polyvalent cations such as calcium, which is advantageous for purposes requiring gel formation. Fourier Transform Infrared Spectroscopy (FTIR) analysis confirmed the presence of functional groups such as O-H (Hydroxyl group), C-H (Hydrocarbon or Alkanes), C≡C (Alkyne), C=O (Carbonyl group), and C=C (Alkene) in Low-Methoxyl Pectin, indicating its functional group. When combined with other ingredients such as polyhydroxybutyrate (PHB), the resin formulation exhibited stability under UV exposure. Even the commercial resin, which showed minor changes in functional groups like O-H and N-H stretching, the resin formulation demonstrated no significant alterations before and after UV exposure. This indicates its resistance to photopolymerization-related degradation and structural instability, making it a suitable candidate for 3D printing applications requiring durability under UV light. The commercial resin demonstrated better photopolymerization under UV exposure compared to the formulated resin. Future optimization of the resin formulation could enhance its performance in UV 3D printing. Adjustments such as incorporating more effective crosslinking agents such as methacrylate and optimizing photoinitiator concentration to improve

photopolymerization and curing efficiency are recommended. Additionally, addressing solvent evaporation rates and conducting rheological tests, such as viscosity analysis, would ensure ideal extrusion properties for 3D printing. These steps could further enhance the potential of this resin formulation for practical applications in UV 3D printing.

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Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: ***study conception and design****: Nur Alia Syamira Zaini, Siti Fatimah Zaharah Muhamad Fuzi;* ***data collection****: Nur Alia Syamira Zaini;* ***analysis and interpretation of results****: Nur Alia Syamira Zaini;* ***draft manuscript preparation****: Nur Alia Syamira Zaini, Siti Fatimah Zaharah Muhamad Fuzi. All authors reviewed the results and approved the final version of the manuscript.*

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