Enhancement of maltodextrin-based adhesive properties using ammonium dihydrogen phosphate (ADP)

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Abstract. Maltodextrin is a new saccharide-based adhesive that can be potentially developed as an alternative for particleboard due to its abundant resources. The addition of ammonium dihydrogen phosphate (ADP) was expected to be able to improve the properties of the maltodextrin, especially the water resistance of the cured adhesive. This study aimed to investigate the properties of maltodextrin/ADP adhesive in the ratios of 100/0, 90/10 and 80/20 wt%. The results showed that the increasing ratio of ADP in maltodextrin-based adhesive can increase not only the insoluble matter rate during boiling condition, but also the other adhesive properties of maltodextrin by lowering the viscosity and increasing the wettability tested in salacca frond particles. The pH adhesive decreased along with the increased ADP ratio. One-way analysis of variance and Tukey test showed that the maltodextrin/ADP ratios significantly affected the adhesive properties. The thermogravimetric analysis (TGA) of dried mixture adhesive showed the significant changes in the onset and the highest weight reduction temperature of maltodextrin after the ADP addition. The FTIR analysis detected some new peaks that were expected to be related to furan ring and carbonyl groups after the maltodextrin/ADP 90/10 and 80/20 wt% were heated at 200°C for 10 minutes and/or 15 minutes. Maltodextrin/ADP 80/20 wt% had the best adhesive properties for particleboard application.

1. Introduction
Currently, massive studies and development of a new natural wood adhesive are conducted due to the limitations/problems of fossil fuel-based adhesive (formaldehyde-based adhesive) in terms of future availability, environment, and human health [1-2]. This led to the discovery of a superior natural adhesive in quality/properties that had high feasibility in the wood industry. This resulted in a substitution use of synthetic adhesive into partly or fully natural wood types. For partial substitution, some studies found that phenol in phenol formaldehyde (PF) can be replaced by tannin, lignin, and cardanol, meanwhile the formaldehyde could be replaced by hydroxymethylfurfural, furfural, and glyoxal [3]. For full substitution, various natural adhesives had been studied such as protein-based (casein, gluten, soy, animal blood, etc), tannin, lignin, citric acid, catechin, and saccharides/carbohydrates-based adhesive (glucomannan, chitosan, gum, starch, dextrin, sucrose, and maltodextrin) [4-11]. It can be used singly or combined with other natural adhesives to improve its properties, e.g. maltodextrin-citric acid adhesive [11].

Maltodextrin has some advantages of being developed to natural wood adhesive due to its abundant availability of the raw material (starch), affordable price, freely soluble in water, and widely available in the market. Maltodextrin can be produced by hydrolysis of various kinds of starch [12]. It has low
water resistance and be combined with citric acid and/or ammonium dihydrogen phosphate (ADP) for better water resistance [11, 13]. The effect of ADP addition on the cured-maltodextrin properties was investigated in previous studies. The ADP addition of 10 wt% in maltodextrin increased the water resistance of the cured adhesive. High heating temperature (220 °C) is required in achieving high water-resistance of the cured adhesive [13], and the ratio of ADP are known to affect the sucrose adhesive properties [10, 14]. The effect of the higher ratio of ADP on the cured maltodextrin properties has not been explored. The effect of ADP ratio on the maltodextrin solution properties (or uncured adhesive properties) has not been explored, and these include viscosity, pH, particles’ wettability, and thermal properties. This information determines the desired adhesive preparation and treatment in achieving the best adhesive spreading and bonding.

2. Methods

2.1. Materials

Maltodextrin (DE 10-15, analytical grade) and ammonium dihydrogen phosphate/ADP (CAS No. 7722-76-1, analytical grade) were provided from Zhucheng Dongxiao Biotechnology Co. Ltd., China, and Merck, Germany, respectively. Distilled water was added as solvent in the preparation of the adhesive solution. Salacca (Salacca zalacca) frond particles (passed through 40 mesh retained 60 mesh in size, 12.3% moisture content) were used in determining the wettability through the adhesive solution.

2.2. Preparation of adhesive solution

The adhesive solution was prepared at 60 wt% concentration and was prepared at maltodextrin/ADP ratios of 100/0, 90/10, and 80/20 wt%. Particular for insoluble matter rate test, maltodextrin/ADP ratios of 95/5 and 85/15 wt% were added. In accelerating for a complete mixing, the mixture was subjected to heating at 80 °C until the maltodextrin and ADP were completely dissolved in the distilled water. The solution was then tested to study the uncured adhesive properties.

2.3. Adhesive solution properties (uncured adhesive properties) test

The adhesive solution properties (uncured adhesive properties) i.e. viscosity, wettability through the adhesive, pH, and thermal properties using thermogravimetry analysis (TGA) were tested. Furthermore, the viscosity of adhesive solution at 28 ± 2 °C (room temperature) and 38 ± 2 °C (warm temperature) was tested using viscometer LV-DV (Brookfield, Massachusetts) and S62 spindle, and the test was conducted in triplicate.

The wettability through the adhesive was measured using the corrected water absorption height (CWAH) method by [15]. It was determined with measuring the adhesive absorption height of the particles that had been stacked up to 50 cm vertically in the glass pipe for 48 h. The stacking particles was ensured to not to be pressed and not to leave any air cavity. The base of the pipe was closed by filter paper to prevent the particles from escaping, but the capillarity was still occurred. The wettability through the adhesive was calculated according to equation 1 in triplicate with the specific volumes of maltodextrin/ADP solutions for 100/0, 90/10 and 80/20 wt% ratios are 0.638, 0.792, and 0.803 g/cm³, respectively.

\[
\text{CWAH (mm)} = h_1 \times \frac{D^2 \pi h_2}{4 w s}
\]

where \(h_1\) is the adhesive absorption height of the particles, \(D\) is the inner diameter of the glass pipe, \(h_2\) is particle height in the glass pipe (mm), \(w\) is oven-dried particles weight and \(s\) is the specific volumes of adhesive solutions.

pH was tested using pH meter SK-620PH (Sato, Japan) in triplicate, and thermogravimetry analysis was conducted using STA-PT 1600 (Linseis, Germany). For thermogravimetry analysis, the adhesive was analyzed in the dried mixture state to study the condition during hot pressing and higher temperatures. The adhesive solution was oven-dried in an aluminum cup thinly at 80 °C for 12 h, then was pulverized and was screened through a 60 mesh screen to produce the dried mixture. The dried mixture was oven-dried at 60 °C for 12 h before thermogravimetry analysis and it was scanned from...
room temperature (± 30 °C) to 400 °C at 10 °C/min heating rate under nitrogen purging at 40 mL/min flow rate.

2.4. Cured adhesive properties test
Cured adhesive properties such as insoluble matter rate and chemical analysis were evaluated, and this referred to the dried mixture that has been subjected to heat treatment. The heating temperature is 160, 180, 200, and 220 °C, and the time is 0, 5, 10 and 15 minutes.

The insoluble matter rate (IMR) was evaluated by measuring the percentage of the remaining weight of the cured adhesive after boiling for 4 h and compared to the initial weight [10]. Three grams of the cured adhesive were boiled for 4 h, filtered in filter paper, oven-dried at 80 °C for 12 h, and weighed. The insoluble matter rate was calculated in triplicate using equation 2.

\[ IMR(\%) = \left( \frac{W_1}{W_2} \right) \times 100 \]  

(2)

where \( W_1 \) is the initial dry weight of the cured adhesive and \( W_2 \) is the insoluble matter remaining weight after oven-drying at 80 °C for 12 h.

The chemical analysis of the cured adhesive was evaluated using Fourier transform infrared spectrophotometer (Shimadzu IR Prestige-21, Japan). The insoluble matter was used as sample to remove the bias of unreacted maltodextrin and ADP in the analysis. It was pulverized to powder size (passed through 100 mesh) and oven-dried at 60 °C for 12 h before the analysis. Kbr disk method was conducted and the analysis was recorded at a resolution of 16 cm\(^{-1}\) with an average of 10 scans.

3. Result and Discussion
The adhesive solution had a clear white color for all maltodextrin/ADP ratios (figure 1a). There was no color change in solution after ADP addition. However, a color change occurred after the solution was oven-dried at 80 °C for 12 h (the dried mixture) (figure 1b). The dried mixture of 100/0 wt% ratio (100% maltodextrin) remained in clear white color, but the ADP addition caused a color change into yellow/brown which indicates occurrence of reaction. It might be the early stage caramelization of maltodextrin since the ADP catalyst hastens the caramelization of carbohydrates [16], and further studies should be conducted.

![Figure 1. a) Maltodextrin/ADP adhesive solution and b) the unpulverized dried mixture.](image_link)

3.1. Adhesive solution properties (uncured adhesive properties)
3.1.1. Viscosity, pH, and particle wettability. Adhesive viscosity is important information used to determine the right spreading method of composite product manufacturing, as well as the integrated step needed to make the production process more effective. Furthermore, viscosity affects the adhesive shelf life, wettability, and the adhesive penetration into wood, so it determines the composite product quality [17-18]. The viscosity of the maltodextrin-based adhesive is shown in table 1, and it ranged from 254.1 to 968 cP. The increased ADP ratio lowered the viscosity of maltodextrin-based adhesive from 23.4 to 44.8%. This was expected to be caused by the lowering percentage of maltodextrin that has molecular weight of 9000 g/mol and replaced with ADP that has a molecular weight almost 9 times smaller, which is 115.02 g/mol [19-20]. It is not due to catalytic reaction between ADP and maltodextrin since ADP was reactive at high temperatures. As the molecular weight decreases, the intermolecular forces (London
force) and interaction become weaker, so molecular mobility increases and the viscosity decreases [21]. Molecular weight, polymer density, and solution temperature are known to affect the viscosity especially Newtonian viscosity as in maltodextrin [22].

The viscosity of maltodextrin-based adhesive at room temperature is still above 500 cP, therefore, it is not suitable to be applied/spread using the spray gun. The viscosity of resin should be 100-500 cP in the particleboard and fiberboard manufacture to ensure that it can be pumped, saved, and spread with high efficiency [23]. The spray is known to be the best spreading method in particleboard/fibreboard production that required low resin viscosity (under 500 cP). Increasing the adhesive solution temperature can lower the viscosity almost 50% for each ratio. The viscosity of maltodextrin-based adhesive at warm conditions is approximately identical with the viscosity of urea formaldehyde (solid content 67.8-69%) that uses spray as a spreading method, i.e. 307-490 cP at 25 °C [24]. Other methods such as dilution (decreasing concentration solution) and the viscosity reducer addition may also lower the viscosity.

**Table 1. Viscosity, pH, and gelation time of maltodextrin-based adhesive.**

| Temperature (°C) | Maltodextrin/ADP (wt%) |
|-----------------|------------------------|
|                 | 100/0 | 90/10 | 80/20 |
| Viscosity (cP)  | 28 ± 2 | 968.0 ± 19.0 f | 741.7 ± 30.6 e | 534.6 ± 9.1 d |
| pH              | 38 ± 2 | 741.7 ± 30.6 e | 534.6 ± 9.1 d | 458.9 ± 32.4 c |
| Wettability (mm)| 28 ± 2 | 341.7 ± 28.5 b | 254.1 ± 8.9 a | 6.06 ± 0.08 c |
|                 |        | 2.38 ± 0.06 b  | 2.85 ± 0.09 a |

Different letters after the value showed significant differences between treatments at P < 0.01

Wettability is a property that showed the compatibility between a wood surface and an adhesive. It affected adhesive absorption, adsorption, penetration, and distribution in the surface of the wood substrate [25] so it affects the bonding quality. The wettability ranged from 36.02-104.40 mm (table 1), and it is identical with the wettability of the inner part of Dahurian Larch (Larix gmelini) wood through fenol adhesive (± 100 mm). However, it is lower than the wettability of Todomatsu (Abies sachalinensis) wood and Karamatsu (Larix leptolepis) wood through phenol adhesive, i.e. ± 200 mm and ± 590-700 mm [26]. The wettability through the adhesive is significantly different between maltodextrin 100% and maltodextrin with ADP. This might be caused by the different adhesive viscosity of both ratios. Figure 2 showed the high correlation between adhesive viscosity and the wettability of particles.

![Figure 2. Correlation between viscosity and particle wettability.](image)

**Figure 2.** Correlation between viscosity and particle wettability.

pH is an adhesive characteristic that needs to be considered due to its effect on the working life/pot life of adhesive, the curing process (through the requirement of pressing time), and the internal bonding of composite products [27]. Furthermore, the pH of the adhesive solutions (table 1) is included in the weak to strong acid category. It is identical with the pH of sucrose/ADP (3.7-6.8), the wood pH at temperate (3.3-6.4), and tropical forest (3.7-8.2) [10, 28]. Therefore, an extreme degradation on
bonded wood and an obstacle in the bonding process because of the adhesive pH are expected not to be occurred. Increasing of the ADP ratio in the maltodextrin-based adhesive decreases the pH. This might caused by the decreased percentage of maltodextrin and the replacement by ADP, which has lower pH. Furthermore, this trend was also found in sucrose/ADP and in another acid-salt catalyst, i.e NH₄Cl addition in PF adhesive [29]. Different trend might be occurred with different based catalyst.

3.1.2. Thermal behavior of the oven-dried mixture
The thermal behavior of oven-dried mixture using TGA analysis is shown in figure 3. The 100% maltodextrin adhesive (100/0 wt%) tends to be stable on heating at room temperature to 280 °C with only 2% weight reduction. The onset temperature of the weight reduction of maltodextrin (100/0) was 280 °C and was lower than the onset temperature of weight reduction of starch, which was 300-310 °C. However, it is higher than the onset temperature of weight reduction of starch acetate made from potato, which was 250 °C [30, 31]. The onset temperature of weight reduction shifted to 200 °C for maltodextrin/ADP 90/10 wt% and 188 °C for maltodextrin/ADP 80/20 wt%. This weight reduction showed a decomposition of maltodextrin. ADP hastens the decomposition of maltodextrin as seen in sucrose/ADP adhesive [10].

![Figure 3. Thermogravimetry analysis of oven-dried mixture.](image)

3.2. Cured adhesive properties
Heating the oven-dried mixture of maltodextrin-based adhesive at various temperatures and times causes a color change of the adhesive into dark color. The higher the heating temperature and the longer the heating time, the darker the color of the mixture. ADP addition hastened the color change.

3.2.1. Insoluble matter rate (IMR). IMR value showed when the adhesive is cured completely and the water resistance of the cured adhesive in boiling treatment [10, 14]. Low IMR value indicated that the adhesive is not cured enough or the adhesive has low water resistance, so it is not suitable for exterior application. The IMR value of maltodextrin-based adhesive ranged in 30-47% for maltodextrin/ADP 100/0 wt% at all heating treatment (160 to 220 °C) as well as all maltodextrin/ADP ratio (100/0-80/20 wt%) at 160 and 180 °C (table 2). A significant increase in IMR value occurred in the maltodextrin/ADP ratio (95/5-80/20 wt%) at 200 and 220 °C heating treatment. This result is in consistent with the finding in TGA that showed the onset weight reduction/decomposition at 200 °C for maltodextrin with ADP addition. Therefore, it was assumed that maltodextrin with ADP addition experienced some decomposition into a high water-resistant substance at 200 °C.
In obtaining more than 80% IMR value, maltodextrin/ADP 90/10 wt% adhesive had to be heated at 220 °C for 10 min and maltodextrin/ADP 80/20 wt% adhesive had to be heated at 200 °C for 10 min. Meanwhile, sucrose/ADP 90/10 wt% adhesive had to be heated at 180 °C for 10 min [10]. It showed that sucrose/ADP required a lower heating temperature than maltodextrin/ADP to produce a high water resistance substance. Maltodextrin (oligosaccharides) has a more complex chemical structure than sucrose (disaccharides), therefore, it may need higher temperature or more catalyst to be decomposed into a high water-resistant substance.

Effect of heating time at 200°C on IMR value was not significantly visible on maltodextrin/ADP 100/0 wt%, but it was visible in maltodextrin/ADP 95/5–80/20 wt% (table 3). Increased heating time can increase the IMR value in maltodextrin with ADP addition but there is no effect on the value without heating. Heating plays an important role on the decomposition and curing reaction of maltodextrin/ADP to be a high water-resistant substance. The increase of heating time from 2 min to 5 min and from 10 min to 15 min did not make a significant increase on IMR value. Furthermore, a significant increase in IMR value of 48-72% occurred when maltodextrin/ADP 85/15 and/or 80/20 wt% were heated from 5 min to 10 min.

Table 2. Insoluble matter rate of the cured adhesive at various maltodextrin/ADP ratios and heating temperatures in 10 minutes heating.

| Maltodextrin/ADP (wt%) | Heating temperature (°C) |
|------------------------|--------------------------|
|                        | 160  | 180  | 200  | 220  |
| 100/0                  | 33.51 ab | 34.32* ab | 34.95* ab | 34.82* ab |
| 95/5                   | 34.77 ab | 43.14 b | 59.56 c | 74.25 de |
| 90/10                  | 30.44 a | 46.79* b | 70.12* cd | 84.60* e |
| 85/15                  | 42.54 b | 47.86 b | 70.74 d | 83.25 e |
| 80/20                  | 32.61 ab | 47.76 b | 82.13 de | 83.69 e |

* has been published at [13], different letters after the value showed significant differences between treatments at P < 0.01

Table 3. Insoluble matter rate of the cured adhesive on various maltodextrin/ADP ratios and heating times at 200°C.

| Maltodextrin/ADP (wt%) | Heating time (min.) |
|------------------------|---------------------|
|                        | 0      | 2      | 5      | 10     | 15     |
| 100/0                  | 29.56* a | 33.21 a | 33.03 a | 34.95* a | 29.89 a |
| 95/5                   | 34.76 a  | 50.95 b | 55.29 bc | 57.02 bc | 76.42 cd |
| 90/10                  | 29.28* a | 55.68 bc | 64.47 c | 70.12* c | 74.97 cd |
| 85/15                  | 38.29 ab | 54.22 bc | 48.72 b | 70.74 cd | 81.17 d  |
| 80/20                  | 30.77 a  | 52.56 b | 49.34 b | 82.13 d | 84.14 d  |

* has been published at [13], different letters after the value showed significant differences between treatments at P < 0.01

3.2.2. Chemical change. Chemical changes of maltodextrin-based adhesive at various maltodextrin/ADP ratios and heating time at 200 °C were analyzed by FTIR spectra (figure 4). There was no chemical change in maltodextrin/ADP 100/0 wt% in 0-15 min heating time at 200 °C. A chemical change may occur at maltodextrin/ADP 90/10 and 80/20 wt% as the absorbance of 1636 cm⁻¹ increased from 5 min heating time to 10 min. Three new peaks also appeared in 15 min heating time i.e. 1705, 1520, and 789 cm⁻¹ that might be attributed to C=O group, C=C group, C-H of furan ring, respectively [10, 32-33]. A small shoulder of those peaks in maltodextrin/ADP 80/20 wt%, 10 min heating time at 200 °C and a small shoulder of 789 cm⁻¹ in maltodextrin/ADP 90/10 wt%, 10 min heating time at 200 °C.
°C were also detected. It was assumed that a decomposition of maltodextrin to a high water resistance containing furan ring may be conducted at 200 °C in 10 min heating time for maltodextrin/ADP 80/20 wt% and it was clearly seen in 15 min heating time for both maltodextrin 90/10 and 80/20 wt%.

![Figure 4. FTIR analysis of maltodextrin-based adhesive at various maltodextrin/ADP ratio and various heating time at 200 °C.](image)

**4. Conclusion**
ADP addition in maltodextrin-based adhesive improved the adhesive properties in both uncured (solution) and cured states for particleboard application. The viscosity and pH were decreased while the wettability in particles increased along with the increased ADP ratio in maltodextrin. ADP addition in
maltodextrin hastened the weight reduction of maltodextrin. Furthermore, increasing the heating temperature and heating time in maltodextrin/ADP (95/5-80/20 wt%) can increase the water resistance of adhesive that might be caused by maltodextrin decomposition into high water resistance substance containing furan ring.

Acknowledgement
The authors are grateful to Ministry of Research, Technology, and Higher Education of the Republic of Indonesia for funding this study under the UGM research grand in 2019-2021.

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