Preparation and Properties of Walnut Cake-based Wood Adhesive with Oxidation Modification

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Walnut cake has the potential for use in preparing wood adhesives because of its richness in protein and carbohydrate. In this work, walnut cakes were treated with sodium periodate or potassium permanganate and then were directly used as wood adhesives. Their bonding properties, curing performances, thermal properties, and chemical structures were compared. The results showed that: (1) The oxidation by $KMnO_4$was non-selective. The reaction was very intense, accompanied by the great variability of oxidation degree and degradation degree, enormous viscosity of oxidation products, high coating difficulty, and low content of active aldehyde groups. (2) The oxidation by $NaIO_4$ was selective; the reaction was mild and easy to control. More active aldehydes could be produced and the treatment was beneficial for constructing a spatial net structure of the adhesive. (3) Compared with oxidation of $KMnO_4$, the walnut cake adhesive prepared by $NaIO_4$ oxidation exhibited a more compact structure, a higher crosslinking degree, low curing temperature, and high thermal stability after curing; its bonding performances met the requirements for Class II plywood specified in GB/T 17657(2013).

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Keywords: Walnut cake; Protein; Carbohydrates; Oxidation reaction; Wood adhesive

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INTRODUCTION

In recent years, soy protein-based adhesives prepared with soybean cake powder after oil extraction have shown great development potential. They have become a research hotspot because of their wide source of raw materials, low price, environmental protection, renewability, and degradability. A lot of research has been conducted around their shortcomings, such as poor water resistance and bonding properties, high viscosity, slow curing speed, and proneness to mold (Lestari *et al*. 2011; Zhang *et al*. 2011; Chen *et al*. 2019; Wang *et al*. 2019; Wu *et al*. 2020; Wei *et al*. 2021; Deng *et al*. 2022; Li *et al*. 2022; Lin *et al*. 2023). Soybean protein molecules are spherical structures that can be further modified only by degrading the spherical protein molecules into chains, exposing hydrophobic groups and active groups, and generating active sites (Li *et al*. 2015; Huang *et al*. 2022; Bai *et al*. 2023). The common method is to cut off the molecular chain by alkaline treatment to expose more active groups, which, however, results in high temperature and is more time consuming in the curing stage.

What is more important and unavoidable is that the problem of "competing with people for food" will be induced if soybean, a main crop variety, is used as raw material to prepare wood adhesives (Kalapathy *et al*. 1996; Wang and Sun 2002; Wu *et al*. 2013; Chang *et al*. 2020; Zhu *et al*. 2024). Hence, it is imperative and significant to prepare wood adhesives using other non-edible vegetable protein-based raw materials.

The research group made a preliminary study on the preparation of wood adhesives from *Jatropha curcas* cake and *Camellia oleifera* cake (Deng *et al*. 2022, 2021, 2023a; Chen *et al*. 2021). The results revealed that the adhesives prepared following the degradation and crosslinking methods of soy protein-based adhesives can meet the national standard requirements for Class II and even Class I plywood, but the adhesives are subject to such problems as uneven degradation, poor initial adhesion, short working life, and large dose of crosslinker. This is mainly because the cake contains a large amount of cellulose and insoluble carbohydrate, which, on the one hand, affect the solubility of protein and the accessibility of alkali. On the other hand, these substances cannot be effectively degraded by alkali and do not participate in the synthetic reaction of adhesives, further influencing their bonding properties.

Frihart *et al.* (2019a, 2019b) introduced a new preparation method of cake-based adhesives. Specifically, soybean cakes oxidized at normal temperature were directly used as wood adhesives to achieve excellent bonding strength. It was found that the dry and wet bonding strength of soybean protein adhesive-based plywood can be significantly improved by periodate, permanganate, or iodate treatments. Meanwhile, the improvement of different oxidants is ranked as permanganate>iodate>nitric acid>chlorate>perchlorate>bromate, among which the dry strength and warm water resistance of adhesive prepared through permanganate oxidation reached as high as 1.7 and 0.9 MPa, respectively. Meanwhile, the researchers applied this method to rapeseed and cottonseed cakes, and the results showed that although the performance of adhesives prepared using these materials is inferior to that of soybean protein adhesives, the universality of the oxidation treatment method for improving the bonding properties of cake-based adhesives was demonstrated. Then, it was further verified by the researchers that the carbohydrates in cakes are oxidized into non-volatile aldehydes, which experienced crosslinking reactions with cake protein to improve the performance of adhesives (Xu *et al*. 2021; Song *et al*. 2023a,b; Yin *et al*. 2024). Therefore, the presence and content of carbohydrates decide the content and structure of aldehyde groups generated, which play a critical crosslinking role in the curing of adhesives.

Walnut is a special industry with distinctive local features and advantages in Guizhou Province. It is estimated that the walnut yield in China will reach 4.9 million tons in 2024. Therefore, in the process of walnut oil extraction, many walnut cake by-products will inevitably be produced. It is reported that the protein content in walnut cake is about 50%, which is equivalent to the protein content of soybean cake. Thus, the material has a natural advantage for developing protein-based adhesives. Especially, the carbohydrate content in walnut cake is about 15%, which is suitable for preparing walnut cake adhesives by oxidation (Xu *et al*. 2015; Wang *et al*. 2019; Jia *et al*. 2020; Malayil *et al*. 2023; Deng *et al*. 2023b). On this basis, the walnut cakes treated by oxidants sodium periodate and potassium permanganate were directly used as wood adhesives in this study. Then, their bonding performances, curing properties, thermal properties, and chemical structures were mainly compared, expecting to provide a new method for the preparation of walnut cake adhesives.

EXPERIMENTAL

Materials

The160-mesh walnut cake powder (protein content of 45.2%, and carbohydrate content of 15.5%) was provided by Guizhou Academy of Forestry. Potassium permanganate ($KMnO_4$, 99.5%), sodium periodate ($NaIO_4$, 99.5%), and sodium hydroxide (NaOH, 99.0%), analytically pure, were provided by Shanghai Macklin Biochemical Co., Ltd. Distilled water was self-made in the laboratory. Poplar veneer (moisture content: 8 to 10%) was obtained from Muyang, Jiangsu, with veneer width of 400 mm×400 mm and thickness of 1.5 mm.

Preparation of Adhesives

A total of 120g of water and 6% NaOH were added into a round-bottomed three-necked flask equipped with a mechanical stirring bar, a thermometer, and a condenser tube. The mixture was quickly stirred and heated to 70 ℃, and 30 g of walnut cake powder was slowly added. This was followed by heat preservation for 60 min. Next, the mixture was cooled to 45 ℃, 12% $NaIO_4$ (proportion in walnut powder) was added, and the mixture was then kept in dark place for the reaction for a certain time. Subsequently, 10 mL of ethylene glycol was added. After reaction for 20 min, the oxidation reaction was terminated, and the mixture was cooled and discharged. The oxidation time was taken as a variable, being 10 min and 20 min, under which the adhesives obtained and were noted as NI-1 and NI-2, respectively. The adhesive prepared with $KMnO_4$ for the reaction for 10 and 20 min were noted as KM-1 and KM-2. The adhesive prepared without adding any oxidant was taken as the blank control group. The aldehyde contents of the prepared adhesive were tested according to the literature (Song *et al*. 2023a).

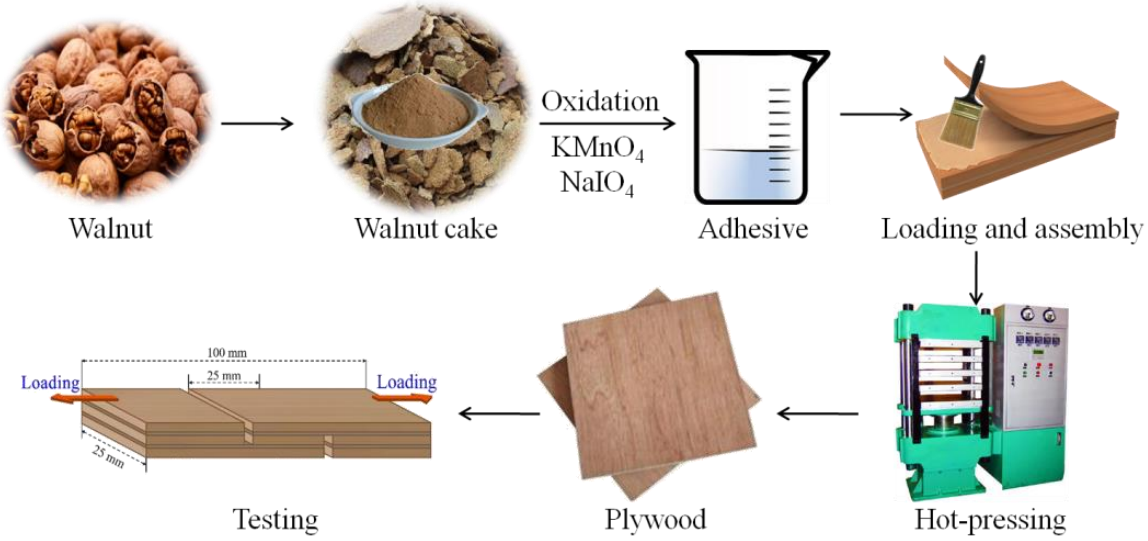

Fig. 1. Diagram of adhesive preparation and plywood preparation and testing

Preparation of Plywood and the Test of Bonding Strength

Three layers of poplar plywood with a width of 400 mm × 400 mm were prepared using the prepared adhesives in the laboratory. The hot-pressing process was implemented under the following conditions: hot pressing temperature of 180 ℃, hot pressing pressure of 1.0 MPa, hot pressing time of 5 min, and adhesive loading of 200 g/m^2. The thickness

compression factor was about 25% for the prepared plywood. The plywood was allowed to stand for two days to release the stress and then cut into specimens with a size of 100 mm × 25 mm, and the shear strength test was performed in accordance with the requirements for Class II plywood in national standard GB/T 17657 (2013). The maximum and minimum values were deleted, and then the rest of the samples were averaged. The standard deviation was less than 5%.

Fourier Transform-Infrared Spectroscopy (FT-IR)

The test was conducted *via* Varian 1000 (Varian, Palo Alto, CA, USA) infrared spectrometer under the following parameters: wave number range of 400to4000 cm^{-1}, resolution of 4 cm^{-1}, and scanning times of 32.

X-ray Photoelectron Spectroscopy (XPS)

The test was performed using an X-ray photoelectron spectroscope (ThermoFischer, Waltham, MA, USA, model ESCALAB 250Xi). The excitation source was ka ray (Al: photon energy *hv*=1486.6 eV); full-spectrum scanning with energy of 100 eV and step size of 1 eV was implemented; narrow-spectrum scanning with pass energy of 50 eV and step size of 0.05 eV was conducted. After the sample to be measured was carved by argon ion for 10 s, all bond energies were calibrated with C1s=284.80eV as the criterion, and then the collected data were analyzed by Advantage software (Thermo Fisher Scientific, Advantage v5.9921, Waltham, MA, USA).

Thermal Property Test

Firstly, the prepared walnut cake adhesive was freeze-dried to remove water and then ground into powder. The curing performance was tested by DSC 204 F1 differential scanning calorimeter produced by Germany Naichi Company. The test conditions were N_2 protection, sample mass 5 to 6 mg, the test temperature range was 30 to 180 ℃, and the heating rate was 10 ℃/min. In addition, the thermal stability of the cured adhesive was tested by a TG 209 F3 thermogravimetric analyzer produced by Germany Naichi Company. The test conditions were N_2 protection, sample mass 5 to 6mg, the test temperature range was 30 to 700 ℃, and the heating rate was 10 ℃/min. Each test was repeated three times.

Particle Size Test

The test was performed using a Mastersizer 2000 laser particle size analyzer (Malvern Instruments Ltd., Malvern, UK). After the test, the volume percentage of the sample in different sub-regions within the range of 0.01 to 10000 nm was obtained.

Scanning Electron Microscopy (SEM)

The cross-section of the adhesive layer after curing was analyzed by scanning electron microscope (SEM, Hitachi S-3400N, Tokyo, Japan) under the accelerating voltage of 12.5 kV and observed after metal spraying.

RESULTS AND DISCUSSION

Bonding Performance Analysis

Figure 2 shows the bonding strength results of walnut cake adhesives. It could be observed that the dry bonding strength of the non-oxidized walnut cake adhesive was 0.46 MPa, without wet bonding strength. After oxidation by $NaIO_4$ for 10min, the dry bonding strength of the walnut cake adhesive increased to 0.96 MPa, with wet bonding strength of 0.40 MPa, which was clearly improved despite the failure to meet the national standard requirement of GB/T 17657(2013). As the oxidation time was lengthened to 20 min, the dry and wet bonding strength of the walnut cake adhesive grew to 1.02 and 1.18 MPa, respectively, both of which could meet the national standard. Therein, the wet bonding strength increased 195%. After $KMnO_4$ oxidation for 10 min, the dry and wet bonding strength of the walnut cake adhesive reached 0.90 and 0.35 MPa, respectively, which reached 0.70 and 0.37 MPa, respectively, at oxidation time of 20 min. Contrastingly, it could be seen that the bonding strength would be reduced if the oxidation time was lengthened, which was opposite to the variation trend of $NaIO_4$ oxidation.

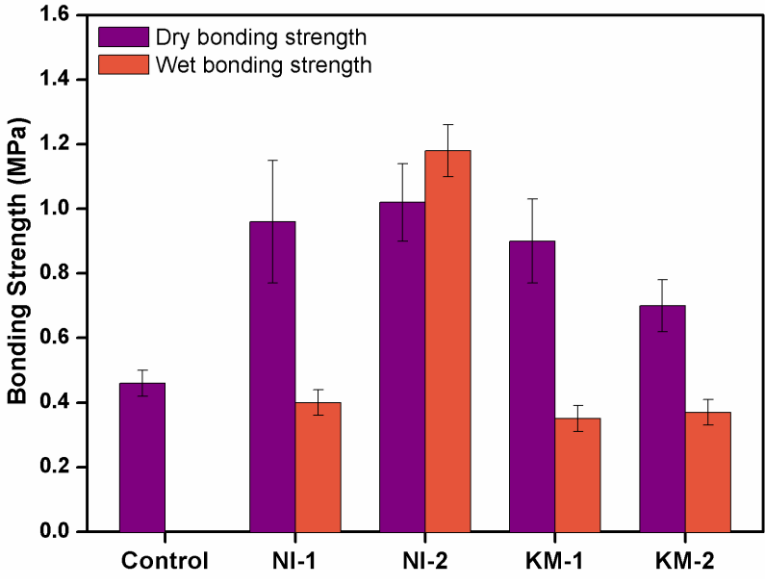

Fig. 2. Bonding strength results of walnut cake adhesives

Oxidation can be divided into selective oxidation and non-selective oxidation (Xiong *et al*. 2023). The oxidation by $KMnO_4$ belongs to non-selective oxidation and it is complicated, during which all hydroxyl groups and unsaturated bonds in the carbohydrate molecular chain may react to obtain complex mixed products, and the oxidation reaction is accompanied by an elimination reaction. So, the aldehyde contents of adhesives prepared with $KMnO_4$ for the reaction for 10 and 20 min were low, with values of 0.51 and 0.35 mmol/g, respectively. The biggest disadvantage is that the oxidation and degradation degrees cannot be controlled, and the oxidation sites and generated functional groups are single and cannot be controlled and determined, which is unfavorable for the preparation of wood adhesives. Therefore, the water resistance of the walnut cake adhesive prepared by $KMnO_4$ oxidation could be improved yet in a limited way.

The aldehyde content of adhesive prepared with $NaIO_4$ for the reaction for 10 and 20 min were 0.83 and 0.96 mmol/g, respectively. That is because $NaIO_4$ oxidation belongs

to selective oxidation, in which primary hydroxyl groups or secondary hydroxyl groups on the carbohydrate molecular chain are directionally oxidated. The reaction is simple and easy to control, which avoids the overall degradation of the oxidation substrate in non-selective oxidation, and the products have high content of aldehyde, which is beneficial to the construction of the spatial net structure of the adhesive. The oxidation product, aldehydes, react with the free amino groups on the protein molecule through Schiff base, which endows the adhesive with good mechanical properties and water resistance, so the walnut cake adhesive prepared by $NaIO_4$ oxidation exhibits good bonding properties. It has been demonstrated that the Schiff base reaction between primary amine and aldehyde group proceeds under mild conditions without by-products, and even the reaction is spontaneous at room temperature (Song *et al*. 2023a,b). This mild reaction condition highlights its simplicity and environmental protection. At the same time, it should be pointed out that the presence and content of carbohydrates determine the content and structure of aldehyde groups, which plays a key crosslinking role in the curing of adhesives.

Figure 3 shows the viscosity results of walnut cake adhesives. It could be seen that the viscosity of the non-oxidized walnut cake adhesive was small, only 20.8 mPa·s. The viscosity of the adhesives obtained through $NaIO_4$ oxidation for 10 and 20 min was 899 and 999 mPa·s, respectively, and that of the adhesives prepared with $KMnO_4$ oxidation for 10 min and 20 min was 8760 and 43,600 mPa·s, respectively. The viscosity change further showed that $NaIO_4$ oxidation was mild, and the viscosity changed little with the lengthening of the oxidation time, and the reaction could be easily controlled. However, $KMnO_4$ oxidation was very intense, and the viscosity increased exponentially with the extension of the oxidation time. Too high viscosity led to poor fluidity of the adhesive, which not only made gluing difficult but also caused the uneven distribution of the adhesive during hot pressing. As a result, the bonding strength of the prepared plywood declined, so the great viscosity was also an important reason leading to the degrading bonding properties of $KMnO_4$-oxidated walnut cake adhesives.

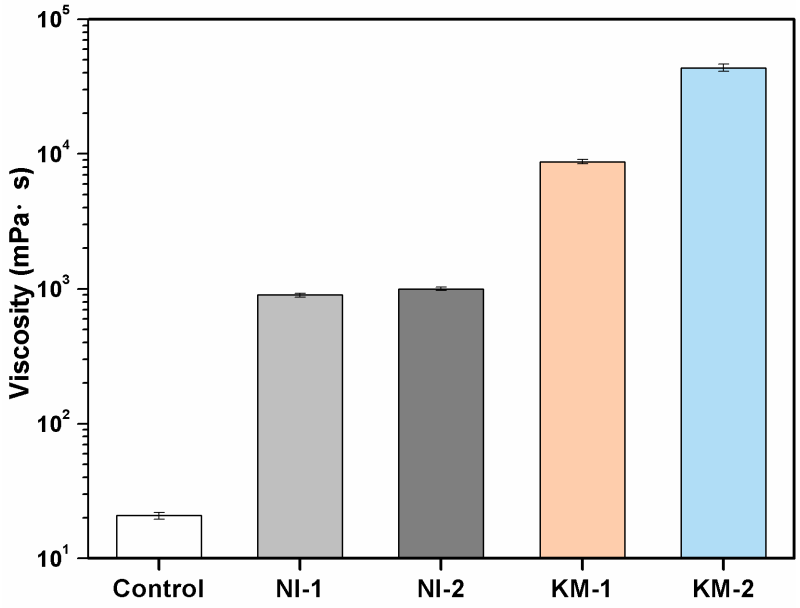

Fig. 3. Viscosities of walnut cake adhesives

Chemical Structure Analysis

Figure 4 shows the FT-IR results of walnut cake adhesives. Walnut cake protein mainly contained groups such as -NH_2, -COOH, and -CONH. The broad absorption peaks with a wave number of about 3400 cm^{-1} were N-H and O-H stretching vibration absorption peaks, and the absorption peak of methylene in protein was at about 2900 cm^{-1}. At 1637.9 cm^{-1} was the C=O stretching peak on the amide bond in the amide I region. Located at 1513.7 cm^{-1} was the N-H bending vibration peak in the amide II region. At 1268.8 cm^{-1} was the C-N stretching vibration peak in the amide III region, and 1378.4 cm^{-1} was the characteristic peak of –COOH. At 1047.2 cm^{-1} was the acetal, disulfide bond or primary alcohol absorption band, and at 879.8 cm^{-1} was the semi-acetal C=O bond.

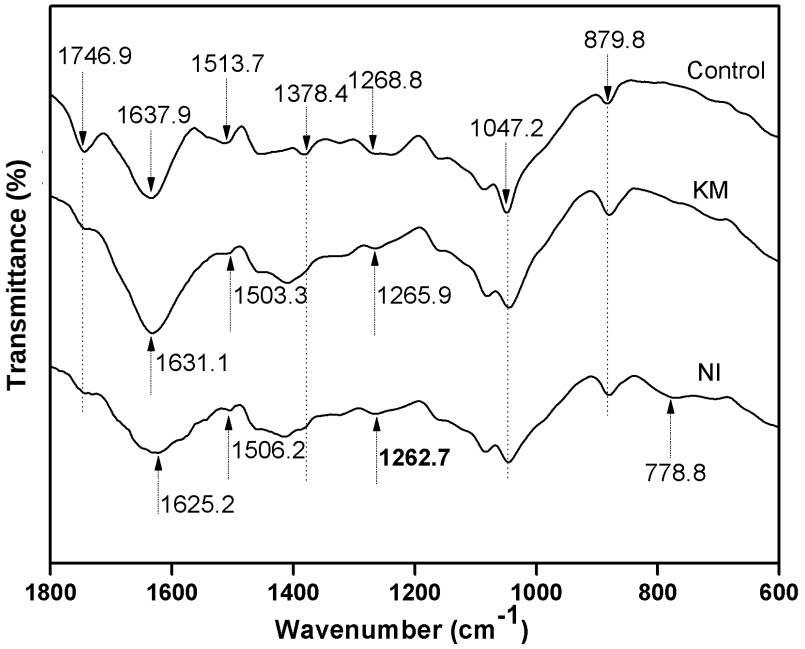

Fig. 4. FT-IR curves of walnut cake adhesives

Through $KMnO_4$ oxidation, the characteristic peaks of amide I, II, and III regions moved to 1631.1, 1503.3, and 1265.9 cm^{-1}, respectively. After $NaIO_4$ oxidation, the characteristic peaks of amide I, II, and III regions moved to 1625.2, 1506.2, and 1262.7 cm^{-1}, respectively, indicating that the obvious blue shift occurred in the amide regions, and the strength decreased to some extent. This might be because the hydroxyl group of carbohydrates in the walnut cake was oxidized to form aldehyde groups or carboxyl groups, and the inductive effect led to the increase in electron cloud density, force constant, and frequency, thus contributing to the blue shift. The blue shift of amide II and III regions of the walnut cake oxidized by $NaIO_4$ was further aggravated, which might be attributed to the higher content of aldehyde groups obtained by oxidation and the denser structure formed by the reaction with protein amino groups, which was reflected in the increased amount of energy needed to complete vibration or stretching in FT-IR spectroscopy.

Figure 5shows the full spectrum and Cls spectrum of X-ray photoelectron spectroscopy for walnut cake adhesives. The C1s diagram of the walnut cake adhesive prepared without oxidation displayed three peaks at 284.8, 285.6, and 289.1 eV, which belonged to C-C, C-O/C-N, and C=O groups, respectively. In the C1s diagram of the adhesives prepared by $NaIO_4$ and $KMnO_4$ oxidation, a new C=N bond was detected at

286.9 eV, and the former content was higher. This further confirmed the FT-IR results, that was the aldehyde groups in oxidation products reacted with the amino group in protein to form the imine bond.

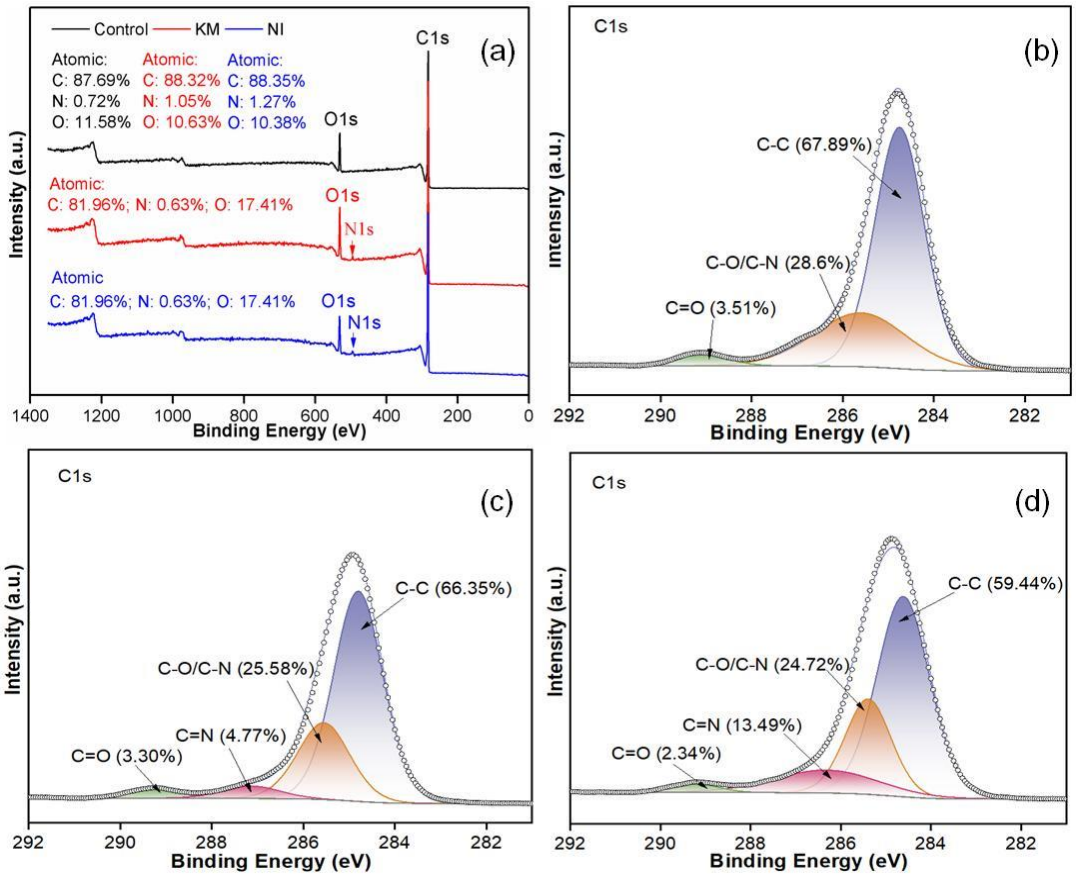

Fig. 5. XPS curves of walnut cake adhesives: a) Full spectrum; b) C1s spectrum of control group; c) C1s spectrum of KM-1 group; d) C1s spectrum of NI-1 group

Particle Size Analysis

Figure 6 shows the particle size of walnut cake adhesives. The peak particle size of unoxidized walnut cake powder was 597.4 nm, while that of the adhesives prepared by $NaIO_4$ oxidation and $KMnO_4$ oxidation was 523.9 and 427.5 nm, respectively, indicating that $KMnO_4$ oxidation destroyed the macromolecular structure of the system (Kang *et al*. 2016; Gu *et al*. 2020; Xu *et al*. 2022), while $NaIO_4$ oxidation had little effect. The non-selective oxidation of $KMnO_4$ led to the excessive destruction of the molecular skeletons of protein and carbohydrate in the walnut cake, which reduced the cohesive strength of the adhesive.

Meanwhile, the content of aldehyde groups in the oxidation product was very small, failing to further strengthen the crosslinking density and stability of the adhesive. The selective oxidation of $NaIO_4$ could not only protect the macromolecular main structure of the protein and carbohydrate in the walnut cake from being destroyed, but also produce more active aldehydes with crosslinking reaction. Moreover, it not only ensured the production of more active groups, *i.e.*, high reactivity, but also prevented the macromolecular main structure of protein from being destroyed, *i.e.*, guaranteeing the high cohesive strength of the adhesive.

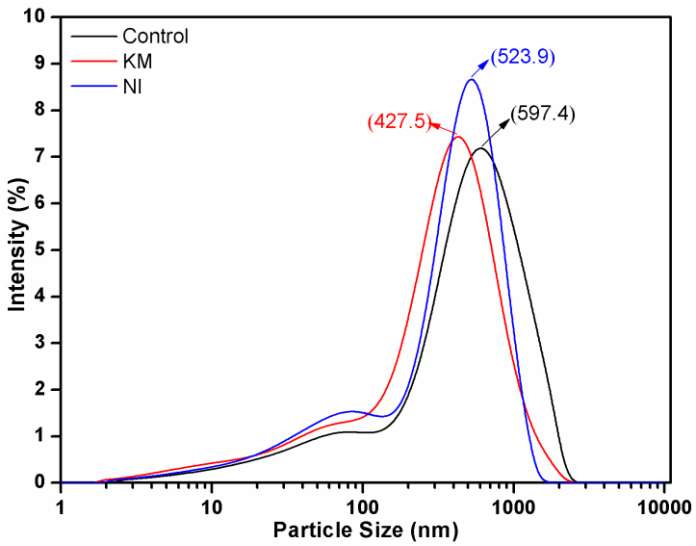

Fig. 6. Particle sizes of walnut cake adhesives

SEM Analysis

Figure7 shows the SEM test results for walnut cake adhesives.

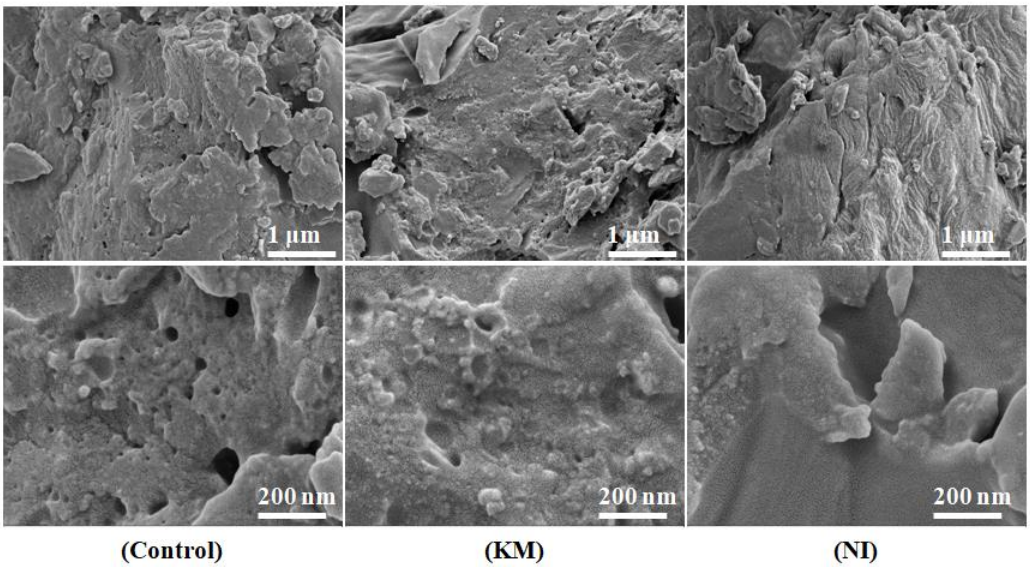

Fig. 7. SEM curves of walnut cake adhesives

The surface of the cured layer of the walnut cake adhesive prepared without oxidation was rough, crisp, and porous. This was because water was easily formed by volatilization due to the lack of crosslinking reaction in the curing of the adhesive, and the cured adhesive layer was also easily immersed by water and lost its water resistance (Deng *et al*. 2023a; Chen *et al*. 2021; Song *et al*. 2023a). The surface compactness of the cured layer of the walnut cake adhesive prepared by $KMnO_4$ and $NaIO_4$ oxidation significantly increased, especially the latter was more compact and smoother, manifesting that the adhesive had a compact structure and a high crosslinking degree after curing. A dense spatial net structure was formed, making it difficult for water to enter the cured adhesive layer, and showing high bonding strength and water resistance.

Thermal Property Analysis

Figure 8 exhibits the curing performance and heat resistance test results of walnut cake adhesives, respectively. It could be observed from Fig. 8 that all three adhesives had a broad exothermic peak at 68.9℃, which was caused by the cleavage of protein disulfide bonds and phase transition of some component.

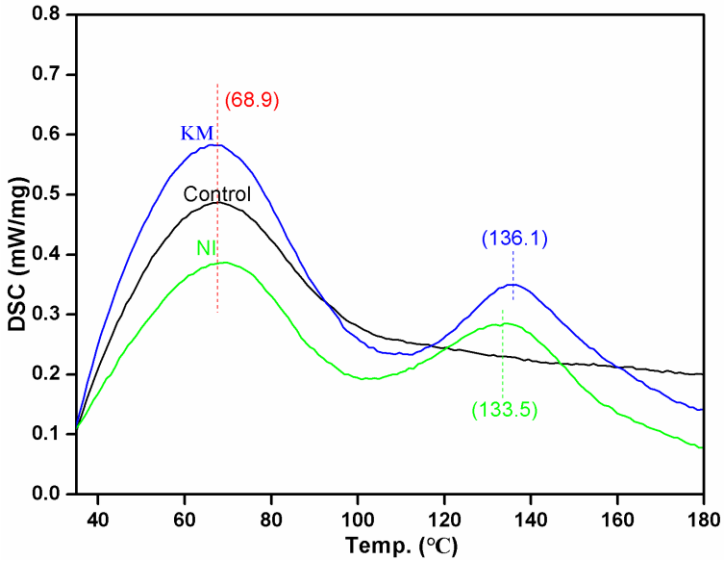

Fig. 8. DSC curves of walnut cake adhesives

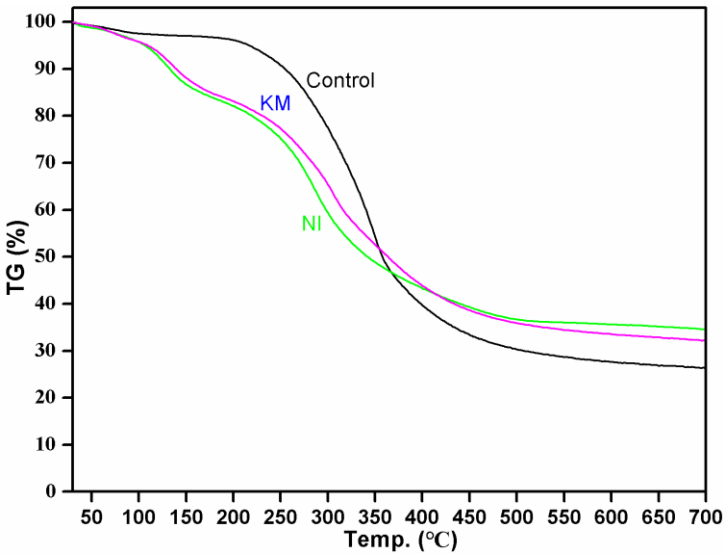

Fig. 9. TG curves of walnut cake adhesives

The crosslinking reaction of the walnut cake adhesive prepared without oxidation was not obvious, so no exothermic peak of the adhesive crosslinking reaction was observed above 100 ℃. The DSC curves of the walnut cake adhesives prepared by $NaIO_4$ oxidation and $KMnO_4$ oxidation showed the exothermic peaks of the crosslinking reaction at 133.5 ℃ and 136.1 ℃, respectively, which were the exothermic peaks of the crosslinking reaction between walnut cake oxidation products and cake protein. The lower peak

temperature of the former further showed that the content of active aldehyde groups in $NaIO_4$ oxidation products was higher, and the crosslinking reaction was easier.

TG curves of cured adhesives are shown in Fig. 9. Mass loss in the 30 to 150 °C stage was mainly caused by water evaporation of adhesives in the adsorption air. The second stage was 150 to 350 °C, in which the mass loss rate was the highest, accompanied by the degradation of protein peptide bonds and the skeleton structure of the adhesive. The carbon residue rate at 700 °C further demonstrated that the crosslinking density and stability of the walnut cake adhesive prepared by $NaIO_4$ oxidation were strengthened.

CONCLUSIONS

Walnut cakes were treated by sodium periodate or potassium permanganate and then were directly used as wood adhesives, and their bonding properties, curing performances, thermal properties, and chemical structures were mainly compared. The results showed that:

1. The oxidation by $KMnO_4$ was non-selective. The reaction was intense, accompanied by the great variability of oxidation degree and degradation degree, enormous viscosity of oxidation products, high coating difficulty, and low content of active aldehyde groups.
2. The oxidation using $NaIO_4$ was of selective, and the reaction was mild and easy to control. More active aldehydes could be produced, which was more beneficial for constructing a spatial net structure of the adhesive.
3. Compared with oxidation using $KMnO_4$, the walnut cake adhesive prepared through $NaIO_4$ oxidation exhibited a more compact structure, a higher crosslinking degree, low curing temperature, and high thermal stability after curing, and its bonding performances met the requirements for Class II plywood specified in GB/T 17657 (2013).
4. The method used in this study is simple, feasible, and can lay a foundation for the preparation of wood adhesives from other cake materials rich in protein and carbohydrate.

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