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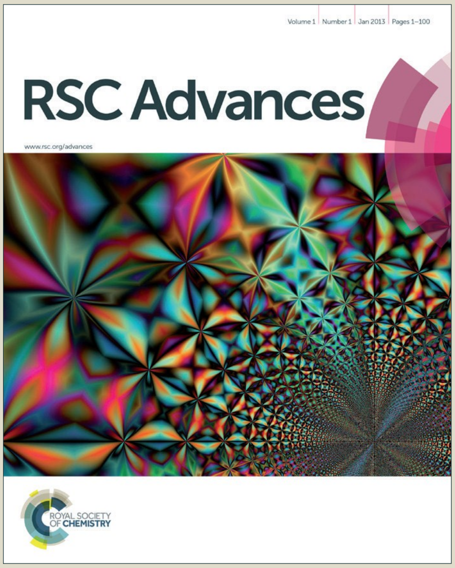

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ARTICLE

Investigating the use of peanut meal: a potential new resource for wood adhesives

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ABSTRACT

This study was conducted to evaluate the potential of peanut meal (PM) to produce plywood adhesives via sodium dodecyl sulfate (SDS) and ethylene glycol diglycidyl ether (EGDE) modification. Five-ply plywood specimens were fabricated to measure water resistance. The physical properties, cross section, thermal behavior, and functional groups of the resultant adhesives were characterized in detail. Results showed using SDS and EGDE in the adhesive formulation greatly improved the water resistance of the resultant adhesive by 90 % and met the interior use plywood requirements. This improvement was attributed to three reason: 1) SDS broke the structure of PM protein and exposed inner active groups, which reacted with EGDE and formed a dense network to improve the water resistance of the resultant adhesive; 2) EGDE decreased the adhesive viscosity and led to the adhesive easily penetrate into wood and forming more interlock; 3) the adhesive with SDS and EGDE created a smooth surface of the cured adhesive to prevent moisture intrusion. Additionally, compared with the peanut meal-based control adhesives, the viscosity of the cured adhesive was reduced by 95.8% to 24,140 mPa•s, which further demonstrated that peanut meal has favorable potential as an alternative adhesive material for plywood.

Introduction

The wood adhesive industry is heavily dependent on petroleum-based chemicals, which are generally favored due to high performance quality and low cost. Said adhesives are non-renewable, and produce formaldehyde emissions, thus posing serious environmental concerns. For this reason, there is growing interest in bio-based adhesives derived from environmentally friendly renewable resources due to their unique characteristics such as biodegradability, renewability, and low ecological impact.[1, 2]

In recent years, soy protein-based adhesive is currently the most commonly researched alternative adhesive.[3, 4] And the mainly disadvantage of the soy protein-based adhesive was the low water resistance. Within the past few years, researchers successfully used synthetized resin to improve the water resistance of the soy protein-based adhesive, such as phenol formaldehyde resin,[5] polyamidoamine epichlorohydrin resins,[6] latex,[7] epoxy resin,[8] and so on. However, all those researches were based on the single resource-soybean meal, which was easily affected by the soy market. And in order to further expand the protein resources, researchers have explored many other plant proteins for wood adhesives, such as wheat gluten and its protein fractions,[9] corn germ protein,[10] and jatropha seed protein.[11] However, the adhesive prepared with the protein was low in these raw materials and the resultant adhesives were not practical for industrial application.

Peanut meal (PM) is a co-product of the extraction of peanut-based edible oil. China has the largest global peanut production industry ever since 1993,[12] largely due to the fact that increased use of peanuts as feedstock for the edible oil industry has likewise increased PM production. Generally, peanut meal was used as livestock feed, fertilizer or fuel, which did not make full use of protein resource.

PM is quite rich in plant protein, with protein content 10-15% higher than soybean meal. The storage proteins of soybean meal are mainly composed of 11S globulins (glycinin), 7S globulins (vicilin), which account for approximately 70% of total storage proteins.[13] Compared to the soy protein, about 87% of the peanut protein is globulin, consisting of two major fractions: arachin (glycinin) and conarachin (vicilin), which is higher than that in soy protein.[14] For soy protein-based adhesive, the 7s and 11s globulins are main mass to produce the bond force in the adhesives, which contain massive active groups burred inside. These active groups can react with the chemical compounds to improve the water resistance of the resultant adhesive. Therefore, PM proteins have many notable features in common with soybean proteins, consequently, it is highly feasible to replace soybean meal with PM to develop wood adhesive– this can broadens the availability of protein sources of raw materials.

In the 1950s, PM as a raw material was firstly used as the wood adhesive, which consisting of hexane-extracted meal, sodium hydroxide, sodium silicate, calcium hydroxide, and carbon disulfide-carbon tetrachloride.[15, 16] In this formulation, the denatured peanut protein molecular chains formed the mutual entanglement structures and hydrogen bonds and produce mechanical properties during the long curing process. However, the hydrogen bonds were easily broken in the wet state, resulting in the poor water resistance of the adhesive. In the past 10 years, only one article has appeared on wood adhesives with peanut protein. Yang used the peanut, soybean, and blood meal protein hydrolyzates, mixing with phenol-formaldehyde (PF), to develop wood adhesive and found using PM with PF resin have a low water resistance.[17] From another perspective of view, in this research, PM was used as a filler to modify PF resin, which was not a protein-based adhesive. With the progress on the soy protein-based adhesive, using PM as a raw material to develop wood adhesive became more feasible.

Considering the property of the PM, SDS used as a denature agent to break the structure of PM protein and expose inner active groups. Then, using EGDE, containing epoxy groups, may react with the exposed hydrophilic groups in protein and form a network to improve the water resistance of the adhesive, which make a feasible way to develop PM-based wood adhesive. In addition, EGDE with low viscosity may reduce the viscosity of the resultant adhesive and make it easily penetrated into wood to produce more interlock, which increase the water resistance and solid content of the adhesive. In this study, peanut meal flour, SDS, and EGDE were used to develop the proposed peanut meal-based adhesive, and five-ply plywood was fabricated to measure the water resistance of the adhesive. The cross section, thermal behavior, and functional groups of the cured adhesives were used to investigate the mechanism of water resistance enhancement and explore the adhesive potential of PM. The primary goals of this study are proving the feasibility of PM as an effective plywood adhesive, adding value to peanut crop production, and promoting the utilization of PM.

Experimental

Materials

Peanut meal (PM) (58.42% peanut protein content, 200 meshes) was obtained from Xiangchi Grain and Oil Company in Shandong Province, China. The amino acid composition of the peanut meal are tested by Hitachi 835-50 Model Automative Amino Acid Analyzer and presented in Table 1. Sodium dodecyl sulfate (SDS) was obtained from Tianjin Chemical Reagent, China. Poplar veneer (15.7 × 15.7 × 0.6 inch, 8% of moisture content) was provided by Wen'an, Hebei Province, China. The ethylene glycol diglycidyl ether (EGDE) was obtained from Guotai Chemical Reagent, China.

Table 1 Quality percentages of amino acids of peanut protein.

Amino acids	wt (%)
Aspartic acid (Asp)	5.04
Threonine (Thr)	1.61
Serine (Ser)	2.28
Glutamic acid (Glu)	8.26
Glycine (Gly)	1.95
Alanine (Ala)	1.99
Valine (Val)	2.03
Isoleucine (Ile)	2.05
Leucine (Leu)	3.50
Tyrosine (Tyr)	1.53
Phenylalanine (Phe)	2.29
Hlstidine (His)	1.76
Lysine (Lys)	2.81
Argnine (Arg)	3.26
Proline (Pro)	2.29

Preparation of peanut meal-based adhesive

For Adhesive A, peanut meal flour was added to water and stirred for 30 minutes at 20 ^{o}C (weight ratio: PM/water=34/66). For Adhesive B, PM, water, and SDS were mixed and stirred for 30 minutes at 20 ^{o}C (weight ratio: PM/water/SDS=33/65/1). For Adhesive C, PM, water, and SDS were mixed and stirred for 10mins, then EGDE was added and further stirred for 30 minutes at 20 ^{o}C (weight ratio: PM/water/SDS/EGDE=22.3/66.7/1/10). For Adhesive D, PM and water was mixed and stirred for 10 minutes, then EGDE was added and further stirred for 30 minutes at 20 ^{o}C (weight ratio: PM/water/EGDE=23.4/66.6/10). For Adhesive E, EGDE was mixed with SDS solution and stirred for 30 minutes at 20 ^{o}C. (weight ratio: SDS/EGDE/water=1/31/68). All the adhesives solid content was 32%.

Preparation of five-ply plywood

Five-ply plywood samples were made under the following conditions: 180 g/m^{2} of glue spreading for a single surface at 120 ^{o}C of hot pressing temperature, 1.0 MPa of hot pressing pressure and 405 s of hot pressing time. After hot pressing, the plywood samples were stored under ambient conditions for at least 24 h before the water resistance was tested.

Water resistance testing

The water resistance of the interior plywood panels (Type Ⅱ plywood) was determined using a three-cycle soak test in accordance with the American National Standard for Hardwood and Decorative Plywood; Hardwood Plywood & Veneer Association; 2004(ANSI/HPVA HP-1). Ten plywood specimens (2 inch ×5 inch) cut from five plywood panels were submerged in water at 24 ± 3 °C for 4 h, and then dried between 49 °C and 52 °C for 19 h with sufficient air circulation to lower the moisture content of specimens to within the range of 4 to 12 percent of the oven-dry weight. All specimens were inspected after the first cycle to determine whether delamination occurred and again after the third cycle, if applicable. This soaking/drying cycle was repeated until three cycles were completed. The criteria for interior application as described in the standard is that 95% of the specimens should not delaminate after the first soaking/drying cycle, and 85% of specimens should not delaminate after the third soaking/drying cycle. A specimen shall be considered as failing when any single delamination between two plies is greater than 2 inches in continuous length, over 0.25 inches in depth at any point, and 0.003 inches in width.

Viscosity

The apparent viscosity of the samples was performed using a HAAKE RS1 rheometer with a parallel plate (P35 35 mm diameter). The distance was set to 1mm for all of the measurements. The experiments were conducted under a steady shear flow at 20 °C. The shear rate dependence of the apparent viscosity was obtained in the range of 1 to 60 s^{-1}. The initial viscosity was selected when the shear rate was 1 s^{-1}.

Scanning electron microscope (SEM) test

The samples were placed in an oven at 120 °C ± 2 °C until a constant weight was obtained. During testing, the surface was sputter coated with gold prior to examining it under the microscope. The fractured surfaces of the coated samples were then examined and imaged using a Hitachi S-3400N (Hitachi Science System, Ibaraki, Japan) scanning electron microscope.

Thermogravimetry (TGA) test

The samples were placed in an oven at 120 °C ± 2 °C until a constant weight was obtained and ground into a powder. The stabilities of the cured adhesives were tested using a TGA instrument (TA Q50, WATERS Company, USA). About 5 mg of the ground powder was weighed in a platinum cup and scanned from room temperature to 600 °C at a heat rate of 10 °C/min. Derivative Thermogravimetry (DTG) means the relation between sample quality change rate and temperature.

Fourier transform infrared (FTIR) spectroscopy test

The samples were placed in an oven at 120 °C ± 2 °C to a constant weight was obtained and ground into a powder. The FTIR spectra of the samples were recorded using a Thermo Nicolet 6700 FTIR over the range of 500 to 4000 cm^{-1} with a 4 cm^{-1} resolution and 32 scans.

Results and discussion

Water resistance

Table 2 The water resistance of the plywood specimens bonded by: (A) the PM adhesive, (B) the PM/SDS adhesive, (C) the PM/SDS/EGDE adhesive, (D) the PM/EGDE adhesive, (E) the SDS/EGDE adhesive,

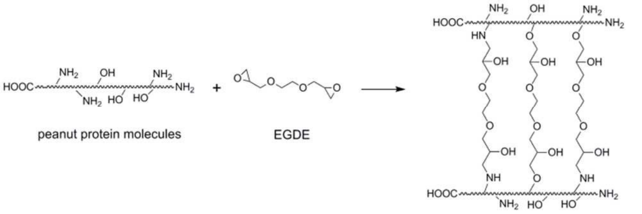
	1st cycle	2st cycle	3st cycle	Pass or Fail
Adhesive A	8/10*	10/10	-	F
Adhesive B	6/10	7/10	9/10	F
Adhesive C	0/10	0/10	0/10	P
Adhesive D	0/10	0/10	3/10	F
Adhesive E	-	-	-	F

*The first number was the number of delamination specimens and the second number was the total testing number of specimens.

Table 2 shows the water resistance of the plywood specimens bonded by different adhesive formulations. Compared to Adhesive A, the water resistance of plywood bonded by Adhesive B increased: one out of ten specimens bonded by adhesive B didn't delaminate after the third cycle. Because SDS is a disperser and denaturing agent known to dissociate and denature protein. The peanut protein molecules in this sample were dispersed and unfolded to where the contact area and the interaction between the wood and peanut protein increased during the curing process, thus improving the water resistance of the plywood. Additionally, the peanut protein contained 15.68% hydrophobic amino acids (Table 1), including Ala, Phe, Ile, Leu, Val, Tyr, and Pro,[18] and after adding SDS, the adhesive was able to break intra-molecular and intermolecular hydrogen bonds, destroy the secondary and tertiary structure of protein molecules, and expose more inner hydrophobic amino acids. These amino acids repelled water while bonding the plywood, ensuring successful interaction between the wood boards and protein adhesives; enhancing water resistance of the plywood in a similar manner as soybean meal-based adhesive.[19] Improvement was limited, however, and did not meet the interior use plywood requirements, because the protein molecules also contained 25.02% hydrophilic amino acids, Arg, Asp, Glx, His, Lys, Ser, and Thr, which were also exposed (Table 1).

Once EGDE was introduced, however (Adhesive C), all ten specimens didn't delaminate after the third cycle, indicating that the water resistance of the bonded plywood significantly increased, which met the interior plywood requirements described in the American National Standard 2000 (ANSI/HPVA HP-1). The EGDE added during the adhesive preparation process reacted with side-chain functional groups of some of the peanut protein amino acids, such as Ph–OH (Tyr), –NH– (primary chain of the peptide and Arg), –C=C– (tryptophan), and –SH (cysteine) and formed several water-resistant chemical bonds during the curing process, which generated network structures that prevented water intrusion and improved cross-linking density, thus enhancing the bond strength of the resultant plywood.[20] Scheme A depicts this reaction in detail. For Adhesive D, three out of ten specimens bonded by the resultant adhesive delaminated and failed to meet the standard requirements for interior plywood, indicating that the utilization of SDS was necessary in this experiment. Additionally, plywood samples were not able to be prepared by

Adhesive E, which was attributed to two reasons: one was its low viscosity, which may over-penetrate an adhesive to the wood's internal structure. The other reason was the difficulty to cure, which should add a curing agent. Therefore, Adhesive E will no longer be discussed.



Scheme A The synthesis procedure for peanut protein with EGDE and its chemical structure.

Apparent viscosity of the adhesives

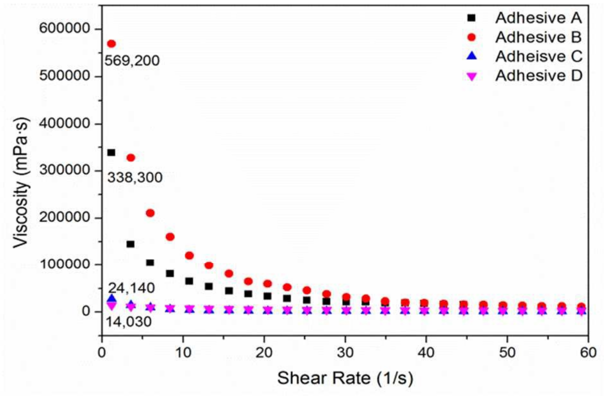


Fig. 1. Apparent viscosity of different adhesive formulations: (A) the PM adhesive, (B) the PM/SDS adhesive, (C) the PM/SDS/EGDE adhesive，(D) the PM/EGDE adhesive.

For any wood adhesive, appropriate adhesive viscosity is a precondition for improved adherence. Viscosity that is too high or too low respectively hinders or over-penetrates an adhesive to the wood's internal structure. In general, favorable operating viscosity limits for the bonding process range between 5000 and 25,000 mPa•s, depending on the application and the nature of the materials to be glued.[21]

The apparent viscosity versus shear rate of different adhesive formulations is shown in Fig. 1. As shear rate increased, the apparent viscosity of all adhesives decreased, until viscosity then grew stable and did not decrease with further increase in shear rate; this demonstrated the shear thinning behavior of the adhesives, defining them as pseudoplastic fluids. For Adhesive A, the highest initial viscosity was 338,300 mPa•s – too high to flow, to where Adhesive A was not able to be applied effectively on the veneer surface, preventing mechanical inter-locking between the wood and adhesive during the curing process and resulting in low shear strength.

The initial viscosity of the Adhesive B increased 68.3%, from 338,300 to 569,200 mPa•s. SDS use increased viscosity by swelling and unfolding protein molecules, decreasing the distance and increasing the friction between them. Moreover, the axial ratio or axis of rotation of protein molecules increased due to swelling and unfolding, further increasing viscosity.[15] Incorporating EGDE dramatically decreased the initial viscosity of the adhesive by 95.8%, from 569,200 to 24,140 mPa•s, because the viscosity of EGDE is very low and the material has small molecular substance – these qualities lubricate protein molecules so that EGDE acts as a disperser, distributed throughout the PM to decrease the viscosity of the resultant adhesive.[22] Compared to Adhesive C, the viscosity of Adhesive D was decreased by 41.9% from 24,140 to 14,030 mPa•s. The result further proofed that the use of SDS improved the viscosity in this experiment.

SEM analysis

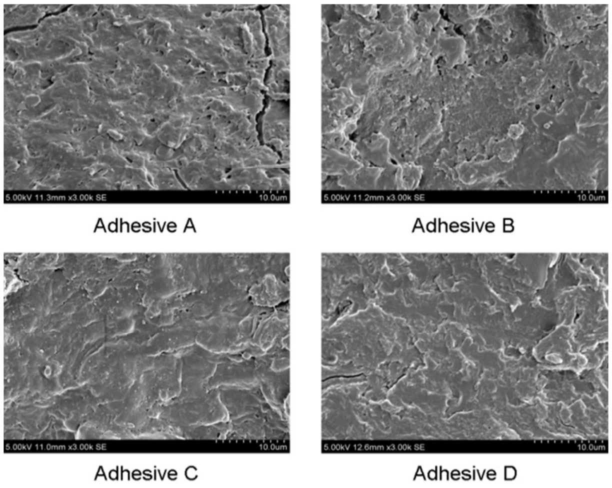


Fig. 2. The cross sections of cured adhesives in different formulations: (A) the PM adhesive, (B) the PM/SDS adhesive, (C) the PM/SDS/EGDE adhesive, (D) the PM/EGDE adhesive.

The morphology of the fracture surface for cured adhesives in different formulations was observed by SEM, as shown in Fig. 2. Holes and cracks were observed on the fracture surface of the sample bonded by Adhesive A, which resembles previous SEM images of soybean meal adhesive.[23] These holes and cracks were caused by the lock of cohesive strength in the adhesive after the curing process. Compared to Adhesive A, the use of SDS resulted in fewer holes and cracks on the fracture surface (Adhesive B), indicating that the use of SDS increased the cohesive strength in the adhesive. This may because SDS could disperse and unfold protein molecules, which made the peanut protein molecules realigned, and led to a smoother and more uniform surface. After EGDE was introduced (Adhesive C), a much smoother and more homogeneous surface free of cracks was observed, indicating that EGDE could cross-link the unfolded protein molecules to form the polymer network structure and the cohesive strength became stronger which led to a more uniform and defect free network. Additionally, the cured adhesive layer became favorably and toughness dense after the addition of EGDE. Compared to Adhesive D, the surface of Adhesive C was also smoother and more homogeneous, indicating that the unfolded protein molecules could be cross-linked by EGDE more efficiently and the cross-linking density of Adhesive C was higher, so that the water resistance of plywood bonded by Adhesive C was better than others.

TG/DTG analysis

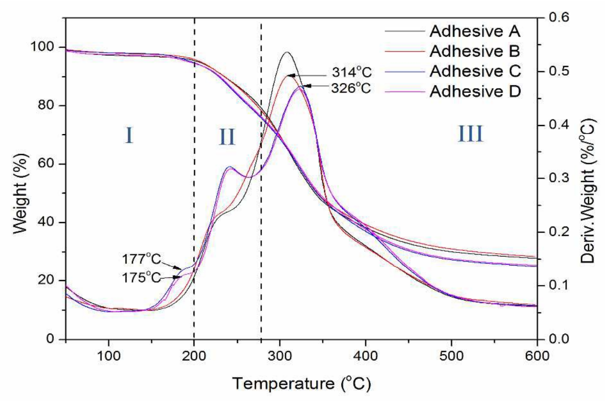


Fig. 3. TGA/DTG curves of different adhesive formulations: (A) the PM adhesive, (B) the PM/SDS adhesive, (C) the PM/SDS/EGDE adhesive，(D) the PM/EGDE adhesive.

Table 3 Thermogravimetric parameters for the thermal degradation of different adhesive formulations: (A) the PM adhesive, (B) the PM/SDS adhesive, (C) the PM/SDS/EGDE adhesive, (D) the PM/EGDE adhesive.

	Adhesive A	Adhesive B	Adhesive C	Adhesive D
T_d* (°C)	176.9	174.5	160.2	167.2

*Initial temperature of degradation.

The thermal behavior of the modified peanut meal-based adhesive was stratified into three main stages (Fig. 3): 50-220 °C (I), 220-285 °C (II) and 285-600 °C (III). In Regions I, the weight loss for the adhesive was attributable to the evaporation of residual moisture. No degradation of soy protein was found and the weight loss ratio was very small. The weight loss in Regions II and III reflected micromolecule loss and unstable chemical bond decomposition[24] and the main skeleton structures of adhesive degradation,[25] respectively.

Wood begins to decompose around 220 °C,[26] Regions I was important for the application of the modified peanut meal-based adhesive. So the thermal behavior of the adhesive would be discussed in Regions I. Comparing Adhesive A with B, the Initial temperature of degradation was decreased from 176.9 to 174.5 °C (Table 3), indicating that the use of SDS decreased the thermal stability of the resultant adhesive, as the same result was gated comparing Adhesive D (167.2 °C) with C (160.2 °C). Because SDS broke intra-molecular and intermolecular hydrogen bonds, and destroyed the secondary and tertiary structure of protein molecules. For Adhesives B and C, the Initial temperature of degradation was decreased from 174.5 to 160.2 °C and a new peak (177 °C) appeared in the DTG curves of Adhesive C blends at Region I, due to further chemical reaction having occurred between EGDE and peanut protein. This chemical reaction could further increase the cross-linking density of the adhesive and improve the water resistance of the resultant plywood, which was in accordance with the result of Luo's research.[20]

In Regions III, the TGA curves may show the property of adhesives with EGDE and side reflect the reason about the increase of the water resistance. So the thermal behavior of the adhesive would also be discussed in Regions III. The main decomposition temperature of blends at Region III significantly increased from 314 to 326 °C between Adhesives B and C, respectively, suggesting that the thermal stability of Adhesive C was better than that of Adhesive B, and likely further implying that the cross-linking reaction between peanut protein and EGDE formed a large amount of chemical bonds and increased the cross-linking density. This analysis provided further evidence that EGDE, SDS, and peanut protein successfully formed high quality adhesives.

FTIR analysis

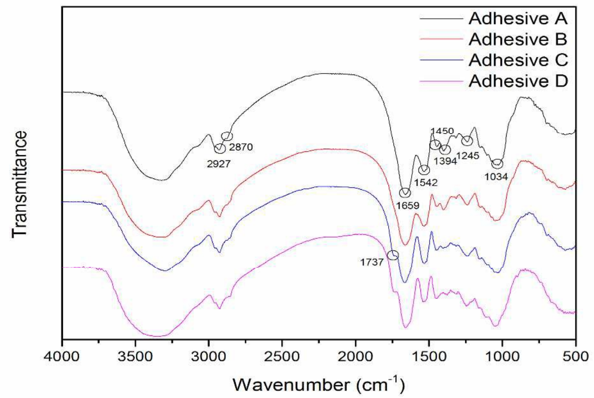


Fig. 4. FTIR spectrums of different adhesive formulations: (A) the PM adhesive, (B) the PM/SDS adhesive, (C) the PM/SDS/EGDE adhesive, (D) the PM/EGDE adhesive.

Table 4 Characteristic infrared bands of peptide linkage.

Wavenumber (cm^{-1})	Description
3600-3000	Free and bound O–H and N–H groups
2927	Symmetric $-NH_2$ stretch
2870	Asymmetric $-NH_2$ stretch
1737	C=O stretch (ester)
1659	C=O stretch, (amide I)
1542	N–H bend, (amide II)
1450	$-CH_2$ (deformation) of methylol groups
1394	COO– bend
1245	C–N stretch, N–H bend, (amide III)
1034	$-C-NH_2$ bend

Fig. 4 and Table 4 show FTIR spectrums of different adhesive formulations. The typical characteristic absorption bands of peptide linkage are related to Amide I, Amide II, and Amide III. Amide I has previously been assigned to α-helix, β-sheet, unordered, and turn areas. Amide II [27, 28] is typically assigned to the symmetrical bending of N–H bonds, a contribution from C–N stretching vibrations, corresponding to the NH^{3+} group of the amino acids. Amide III arose primarily from N–H bending and C–N stretching vibrations with weak contributions from C–C stretching and C=O in-plane bending.[29]

Compared to Adhesive A, there was no new peak in the FTIR spectra of Adhesive B, and a same result was observed between Adhesive C and D, implying that the interaction was only a protein denaturation process rather than a chemical reaction. This observation is consistent with previous research.[23] For Adhesive C, the absorption peak areas of COO– (1394 cm^{-1})[30] dropped lower than the peak at 1450 cm^{-1} of Adhesive B,[31] likely because the epoxy groups of EGDE resin connected with the COO– groups of peanut protein during the curing process, which suggest that the amount of hydrophilic groups in the adhesive decreased after introducing the EGDE, altogether improving the water resistance of the adhesive. A new peak was observed at 1737 cm^{-1}, attributed to the stretching vibrations of ester C=O. This peak indicates that the epoxy group in the EGDE reacted with the carboxyl group of the peanut protein, forming ester linkage. The decrease of hydrophilic groups (peanut protein) and the formation of cross-linking structures (Adhesive C) provide evidence that further proves that PM can, in fact, be utilized successfully to prepare protein adhesives.

Conclusions

The most notable conclusions of this study can be summarized as follows.

Using SDS to formulate adhesive increased the water resistance of the resultant plywood by 10 %, attributing to the protein molecules denaturation and inner hydrophobic amino acids exposition.

Using EGDE reacted with the exposed active groups of the peanut protein molecules and formed additional network structures, markedly improving the cross-linking density of the resultant adhesive by 90%. In addition, introducing EGDE created a smooth surface of the cured adhesive to prevent moisture intrusion and reduced adhesive viscosity to form more interlock with wood surface, which further improved the water resistance of the adhesive.

The viscosity of the PM/SDS/EGDE adhesive was reduced by 95.8% to 24,140 mPa•s and the water resistance of its resultant plywood met interior use plywood requirements, proving that peanut meal has the potential to be utilized readily and successfully to prepare bio-based adhesives.

Acknowledgements

The authors are grateful for Beijing Natural Science Foundation (2151003) and Special Fund for Forest Scientific Research in the Public Welfare (201404501).

Notes and references

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Figure captions

Fig. 1. Apparent viscosity of different adhesive formulations: (A) the PM adhesive, (B) the PM/SDS adhesive, (C) the PM/SDS/EGDE adhesive，(D) the PM/EGDE adhesive.

Fig. 2. The cross sections of cured adhesives in different formulations: (A) the PM adhesive, (B) the PM/SDS adhesive, (C) the PM/SDS/EGDE adhesive, (D) the PM/EGDE adhesive.

Fig. 3. TG/DTG curves of different adhesive formulations: (A) the PM adhesive, (B) the PM/SDS adhesive, (C) the PM/SDS/EGDE adhesive，(D) the PM/EGDE adhesive.

Fig. 4. FTIR spectrums of different adhesive formulations: (A) the PM adhesive, (B) the PM/SDS adhesive, (C) the PM/SDS/EGDE adhesive, (D) the PM/EGDE adhesive.

Table captions

Table 1 Quality percentages of amino acids of peanut protein.

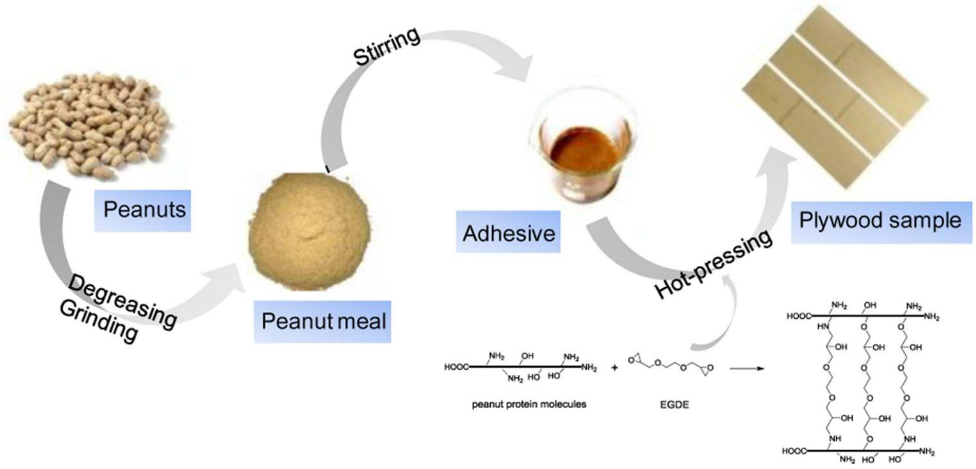
Table 2 The water resistance of the plywood specimens bonded by: (A) the PM adhesive, (B) the PM/SDS adhesive, (C) the PM/SDS/EGDE adhesive, (D) the PM/EGDE adhesive, (E) the SDS/EGDE adhesive.

Table 3 Thermogravimetric parameters for the thermal degradation of different adhesive formulations: (A) the PM adhesive, (B) the PM/SDS adhesive, (C) the PM/SDS/EGDE adhesive, (D) the PM/EGDE adhesive.

Table 4 Characteristic infrared bands of peptide linkage.

Scheme caption

Scheme A The synthesis procedure for peanut protein with EGDE and its chemical structure.



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