



ORIGINAL ARTICLE

Wood Adhesives Derivation from Tannery Waste Protein: A Comparison with some Commercial Wood Adhesives

*Omm-e-Hany¹, Syed Shahid Shaukat¹, Beena Zehra,^{1,2} Aamir Alamgir¹ and Asia Neelam¹

¹Institute of Environmental Studies, University of Karachi, Karachi 75270, Pakistan.

²Leather Research Centre, PCSIR, D-102, South Avenue, SITE, Karachi, Pakistan.

*Corresponding author: (hany786@yahoo.com)

ABSTRACT

In this study, a wood adhesive was prepared from the waste protein isolated from the alkaline hydrolysis of skin flashings wastes. This protein was isolated by alkaline treatment of skin flashings using alkaline mixture of magnesium oxide and potassium hydroxide. The wood adhesive was prepared by blended protein with soluble starch solution and ortho-phosphoric acid and the prepared adhesive formulation was applied in wood samples (Ply wood, Formica, Chip board) in different sizes. The effect of steam from boiling water on joined various wood samples was observed for 2h, 4h and 8h. The results were compared with 3 commercial wood adhesives which are used in local market. It was found that the shear strength of the wood samples was influenced by the steam after a period of 8h. Results were found comparable with the commercial adhesive. Thus, a waste protein recovered from the skin flashings can be utilized as a wood adhesive preparation after suitable chemical modification. The change in thickness of wood samples after 2h, 4h and 8h was evaluated by using ANOVA followed by Duncans Multiple Range test. Significant differences between the prepared adhesive and commercial adhesives were noted. The adhesive prepared in this study provides a good and cheaper alternative.

Key words: Skin flashings, protein, wood adhesive, comparison

Received 04.02.2015

Revised 16.03.2015

Accepted 04.04.2015

INTRODUCTION

Wood adhesives are needed for the manufacturing of wood composites. The early civilizations have already used natural adhesives in decoration of furniture and musical instrument assembly [1]. Natural materials such as proteins are potential replacements for synthetic resins [2,3]. Proteins have been used from previous periods to prepare adhesives, while petroleum-based adhesives started to dominate the market for decades due to their affordable cost and satisfactory performance [4]. However, limited reserves of petroleum and concerns about emission of volatile organic compounds (especially formaldehyde, a carcinogenic compound from petroleum-based adhesives) have recharged attempts to develop bio based adhesives from renewable resources [5,6]. Adhesives prepared from soy bean proteins, animal blood, gluten, and sorghum proteins have also been reported extensively [6,7,8]. As a natural adhesive, soybean flour has been used in the largest volume, it was ground into soybean meal, the residue after the soybeans have traditionally more valuable oil which was removed. The soyabean flour was finely ground and processed through a number of steps to disperse the meal and denature the protein [1]. In many cases, the denatured protein has to be used within eight hours before the adhesive starts to degrade. Soybean protein adhesives were allowed for the development of the interior plywood industry in the early 1900s. The soy bean adhesives have been improved to give better water resistance but never achieved sufficient moisture resistance to make exterior grade ply wood. Phenol-formaldehyde (PF) resins have been measured to displace soy bean adhesives due to cost and insignificant performance. Today, soybean resins are used at some places but these types of adhesives are more often used in small amounts as an additive for the synthetic resins adhesives. The expansion in soybean adhesives was during the 1950s shows the potential for soybean adhesives on a cost basis if the water resistance short storage stability and variation of properties can be overcome. With the rising cost of petroleum based adhesives, soy flour based adhesives are again being studied [9]. Moreover, none of the other protein sources are available in the work places with adequately low cost, large supply and regular composition

as soybean flour but they still have advantages because of their special properties. Blood protein from beef and hogs has the best water resistance of some of the commercial protein adhesives but has great variation [1]. Animal bone and hide glues are used in fine furniture manufacturing since they provide flexible bonds for good stability with internal humidity changes [10]. They have many other uses but are being replaced by synthetics such as ethylene vinyl acetate polymers due to cost and the synthetics greater ability to be formulated for specific applications. Casein, like many of the protein adhesives provides good fire resistance and it is, therefore, used in fire doors. Each of these adhesives has its own process for denaturization and utilization [1].

The solid waste from tanneries is dumped out and creating toxic pollution problems. In the tanneries, there has been increasing emphasis on its planned progress aimed at optimum utilization of available raw materials to maximize the returns particularly from exports [11]. Although, recovery of protein from different leather wastes has been carried out successfully and utilization methodologies have been discussed earlier but some challenges still exist because these wastes from tanneries are potentially difficult from other categories of solid wastes due to the presence of different chemicals which are generally used in leather manufacturing [12].

The products from leather wastes through recycling are sold in the market in the form of binders from trimming wastes, fertilizers from sludge, fatliquors for leather processing at fatliquoring stage, detergents, binders from trimming wastes for drug delivery and wound dressing in clinical applications, generation of bioenergy, poultry and other animals feed, etc. The skin fleshings proteins have been found effective as glue, edible gelatin preparation and dog chew making toys [12]. However, a huge amount of proteinaceous waste is reusable in various useful products after some modification [13]. Therefore, environmental friendly products from leather solid wastes have gained urgent investigations in this field. The objectives of the present study were 1) The isolation of protein from skin fleshings 2) conversion of protein into wood adhesive 3) Application of prepared adhesives as a total replacement of synthetic commercial adhesive product 4) comparison of results through application of commercial adhesives.

MATERIAL AND METHODS

The skin fleshings were collected from the tanneries of SITE, Karachi. These skin fleshings were treated with alkaline solution of sodium hydroxide and magnesium oxide to hydrolyze into valuable fractions in autoclave at 95-110°C for 2h. Different types of wood samples were taken from the local wood market, Karachi. Soluble starch was used from Sigma, Aldrich Germany. Potassium hydroxide was purchased from Merck. Three different wood glues were purchased from the market MOWILITH 270 German glue manufactured by Clariant Pakistan limited, Karachi. It was a synthetic glue used for the adhesion of paper, chip board, hard board and useful for various types of wood such as ply wood. Second was KAYLITH 270 German white glue manufactured by MALCOM Industries, Karachi. It is also used for the adhesion of wood, formica, plywood and chip board. Third was WOODFIT 270 German White glue manufactured by polymer International (PVT) , LTD , Korangi Industrial Area, Karachi. It is used for the adhesion of wood, ply wood, chip board and formica, etc.

Preparation of wood adhesive

To prepare, 100g of wood adhesive, soluble starch solution was prepared in distilled water 20 (p/v) concentration by stirring using magnetic stirrer while slightly warmed at 60°C for 10 minutes. Then, this starch solution was cooled at room temperature. The protein isolated from skin flashings having sufficient amount of moisture (40%) was taken (70 p/v) and starch solution (40ml) was taken. First, protein was heated at 60°C then starch solution was added and 0.5% ortho-phosphoric acid was also added in a flask. After addition of reactants, the pH of adhesive was adjusted to 10-11 by adding potassium hydroxide solution which was prepared by dissolving 5g in 100ml of distilled water. The reaction was proceeded at 60°C with constant stirring for 30 minutes until a viscous adhesive was formed. The adhesive was then cooled at room temperature and stored in an air tight bottle till application.

Application of Prepared adhesive on Wood Panels

The wood samples in different types were prepared in the sizes as given specification in Tables 1 to 4. These samples were cleaned with the fine cloth to remove any dust particles from the surface. These wood samples were tested with some modification adopting the test reported by [4] for the bonded wood composites. Two indicators of performance were available from the test. First, a bonded wood composite might fall apart in this rigorous hot water test such that the two wood strips became unattached. Thus, the % wood strips that remained attached were a measure of the tolerance of the adhesive to hot water. Secondly, for the wood composites where the wood strips remained attached. The prepared adhesive formulation and three commercial wood adhesive were applied only one side of the each piece. These samples were prepared in duplicate with exactly same dimensions to make a bond after

application of adhesive. These samples were dried at room temperature for 5 minutes. Then, these samples were joined and pressed with a weight (10 kg) for 24 h. Next day, these samples were tested for adhesion by the effect of steam of hot water. These samples were hanged at the frontage of steam in an iron wire from a hole in the middle of each sample. Thickness of each type of wood samples was taken according to the (SLP 4, IUP4; BS 3144:method 3) with the calibrated thickness gauge.

RESULTS

It has been studied earlier that the composition of major components of woods vary little from wood to wood, this variation in adhesive strength with the type of wood may be due to the variation in physical properties of woods, such as porosity and degree of surface roughness [14]. Kaylith adhesive was applied on four different types of wood samples. Mean from three replicates of each sample specification is presented in Table 1. The change in each sample thickness was observed after 2h, 4h and 8h. The results are also presented in Table 1. These results show that the highest change in thickness was observed after 8h in the sample type 4 followed by sample type 2 and 3. While lowest change was observed in sample type 1. Commercial adhesive Wood fit was also applied in the same manner. The results are presented in Table 2. The results show that the highest change in thickness was observed after 8h in the sample type 1, followed by sample 3. While lowest change was observed in the sample type 2 and 4 which show similar value. Factorial ANOVA showed that the 4 glue types (treatment 1 to 4) were significant ($p < 0.001$) while time of exposure to steam was also significant ($p < 0.001$). The interaction of glue type x time was also significant (Table 5A, 5B).

Table 1 : Application of adhesive on different wood samples (Treatment 1)

Sample Type	Adhesive Applied	Length(mm)	Width (mm)	Thickness (mm) Before application	Thickness (mm) after 2h	Thickness (mm) after 4h	Thickness (mm) after 8 h	Condition
Ply to Ply	Kaylith	85±8.66*	38.00±0.00	8.7±0.17	8.7±0.17	8.9±0.17	7.66±0.57	Not separated
Chip board to Chip Board	Kaylith	75±0.57	40±0.00	33±2.645	32±2.081	33.33±20.81	31.66±1.527	Separated by force
Wood Formica to Wood Formica	Kaylith	42.66±6.429	40±0.00	30±0.00	30±0.00	31±1.00	29.66±0.577	Not separated
Wood to Wood	Kaylith	41.0±1.732	30±10.0	24±5.291	25±7.0	21.66±2.88	19.0±1.0	Not separated

*standard deviation is given against each result calculated from three observations of each test

Table 2: Application of adhesive on different wood samples (Treatment 2)

Sample Type	Adhesive Applied	Length(mm)	Width (mm)	Thickness (mm) Before Application	Thickness (mm) after 2h	Thickness (mm) after 4h	Thickness (mm) after 8 h	Condition
Ply to Ply	Wood Fit	83.33±5.77*	39.66±0.57	9.0±0.00	9.0±0.00	8.0±0.00	8.0±0.00	Not separated
Chip board to Chip Board	Wood Fit	47±1.0	40±0.00	33.66±1.52	34±1.73	34.33±2.081	34.66±2.309	Not separated
Wood Formica to Wood Formica	Wood Fit	43.33±5.77	38.66±2.309	30±0.00	30±0.00	30±0.00	29.66±0.57	Not separated
Wood to Wood	Wood Fit	42±1.73	32±6.982	24.66±5.03	24.66±5.03	25.33±5.131	25.00±4.0	Not separated

*standard deviation is given against each result calculated from three observations of each test

Mowilith adhesive was applied in the four different types of wood samples. These samples specification is presented in Table 3. The change in thickness was observed after 2h, 4h and 8h. These results are presented in Table 3. These results show that the highest change in thickness was observed after 8 h in the sample type 1 followed by sample type 3 and 4. While lowest was observed in sample type 2. Protein based adhesive was applied in the same four different wood samples. These sample specifications are presented in Table 4. The change in thickness was observed after 2h, 4h and 8h. These results are presented in Table 4. The results show that the highest change in thickness(mm) was observed in sample type 1 followed by sample type 4 While lowest was observed in sample type 2 and 3 which show similar change in thickness.

Table 3: Application of adhesive on different wood samples (Treatment 3)

Sample Type	Adhesive Applied	Length(mm)	Width (mm)	Thickness (mm) Before application	Thickness (mm) after 2h	Thickness (mm) after 4h	Thickness (mm) after 8 h	Condition
Ply to Ply	Mowilith	86.66±4.16*	44.00±6.0 82	8.76±0.152	10.6±0.360	9.00±0.0 0	7.66±0.5 7	Not Separated
Chip board to Chip Board	Mowilith	40±0.00	40.66±0.5 7	36.66±0.57	35.0±0.00	35.0±0.00	35.0±0.00	Not separated
Wood Formica to Wood Formica	Mowilith	50.33±0.577	40.000.00	33.33±1.154	30.66±1.154	30.66±1.154	31.33±1.52	Not Separated but a gap occurred between pieces
Wood to Wood	Mowilith	47.66±5.773	29.33±4.9 32	28.00±7.211	22.33±0.57	22.33±0.57	21.66±2.081	Not separated

*standard deviation is given against each result calculated from three observations of each test

Table 4 : Application of adhesive on different wood samples(Treatment 4)

Sample Type	Adhesive Applied	Length (mm)	Width (mm)	Thickness(mm) Before application	Thickness (mm) after 2h	Thickness (mm) after 4h	Thickness (mm) after 8 h	Condition
Ply to Ply	Protein adhesive	85±4.00	40±0.00	9.1±0.00*	9.1±0.10	9.1±0.10	8.0±0.00	Not separated
Chip board to Chip Board	Protein adhesive	50±5.0	40±0.00	35±0.00	35±0.57	35.33±0.57	35.66±0.57	Separated by force
Wood Formica to Wood Formica	Protein adhesive	46.66±11. 547	46.66±11 .547	30.00±0.00	30.00±0.00	30.33±0.57	30.00±0.00	Not separated
Wood to Wood	Protein adhesive	69.00±13. 892	35.33±8. 962	11.8±2.77	11.8±2.77	11.8±2.77	12.2±3.08	Separated

*standard deviation is given against each result calculated from three observations of each test

Table 5 A : Two Way ANOVA Completely Randomized for Adhesive Effect

Source	SS	df	MS	F	P
Main Effects (time)	20.865	3	6.955	15.734	0.001
(glue type)	45.9613	3	15.297	34.605	0.001
Interaction time xglue type	33.695	9	3.743	8.469	0.001
Error	14.1454	32	0.442		
Total	114.597	47			

Table 5 B : Duncan's Multiple Range Test for Time of wood samples

Error Mean Square = 0.44
Degree of Freedom = 32
Significance Level = 0.05
LSD 0.05 = 0.55

Rank	Treatment No.	Mean	n	Non-Significant Ranges
1	4	2.285	12	a
2	2	0.96	12	b
3	3	0.784	12	b
4	1	0.618	12	b

Table 5 C : Duncan's Multiple Range Test for Glue Type

Error Mean Square = 0.44
Degree of Freedom = 32
Significance Level = 0.05
LSD 0.05 = 0.55

Rank	Treatment No.	Mean	n	Non-Significant Ranges
1	3	2.77	12	a
2	1	1.13	12	b
3	2	0.446	12	c
4	4	0.31	12	c

DISCUSSION

The alkaline hydrolysis of tannery wastes generates amino acids rich liquid hydrolyzed protein [15,16,17]. However, functional properties of extracted protein may be affected by pH, concentration of salt and or other chemical modifications. In the previous studies, protein adhesiveness to the substrate has been studied and it was found that the various amino acids such as glutamic acid, tyrosine, proline, etc. contribute to this adhesive action via hydrogen bonds [18,19]. The adhesive strength of protein glue depends on its ability to disperse in water and on the interaction of polar and non-polar groups of the protein with wood material. In a native protein, the majority of polar and non-polar groups are unavailable due to the internal bonds resulting from van der Waals forces, hydrogen bonds and hydrophobic interactions. For this reason a simple protein is a poor adhesive and a chemical change is required to break the internal bonds and uncoil or disperse the polar protein molecules. An earlier study described that the alkaline treatment (pH10.0) and heating at moderate temperature (40–50°C) improved the adhesive strength of protein, unfold the protein and expose both non polar and polar groups, thus leading to better interaction with the substrate [20]. Like most of the biomass materials, proteins are not uniform in composition as the source of the protein varies, thus, the processes for using these proteins and the properties of the adhesives vary as the protein source changes. The main method of denaturation for adhesive applications is hot aqueous conditions [1]. The aqueous process is often done under alkaline conditions and may also involve adding other chemicals to either stabilize the denatured protein adhesive or add strength to the final bonds. The adhesive was prepared from the skin fleshing recovered protein in a light brown color in a viscous form that was applied easily. The result for adhesiveness was found satisfactory which may be due to the following reasons.

1) An important property of animal protein adhesives which even not in an emulsion form, they are able to form gels which involves intra and inter-molecular rearrangements on cooling, thus providing an immediate strong bond to wood. Further drying can provide final resilient strong bonds with the wood surface [10, 21, 22].

2) Another property of the animal protein adhesives besides their bonding characteristic is their film forming property. The film forming property is a consequence of the use of these protein materials in emulsions form rather than in gel form. However, in the emulsion form, these proteins can not be used suitably but need a modification strategy in order to improve the final adhesive properties. This was mostly achieved by blending them with different chemicals such as glycerine, sorbitol, and other glycols, sometimes with mineral fillers to increase the film flexibility property. Thereafter, these adhesives are usually graded according to the viscosity and the gel strength [10, 21, 22]. The main components of wood are cellulose, hemicellulose, and lignin. Hemicellulose is the fraction most accessible to water and is reported to be the main site of interactions (hydrogen bonding, van der Waals force) between wood and water [23]. Molecules in the adhesive must come into direct contact with the molecules in the wood to provide the best mechanical interlock and intermolecular attractions between adhesive and wood. The adhesive strength trends to decrease by the presence of air-filled voids, defects, or geometrical features in the wood that cause fractures in the joint under stress. The contacts are induced by curing, a process leading to loss of solvent in the adhesive (due to evaporation of solvent from the adhesive and diffusion of adhesive into the wood) and to produce linkages between polypeptide chains at elevated temperatures. It has been proposed that the bonds between wood and adhesive are mainly hydrogen bond and van der Waals force [24]. Like the most polymers, proteins become harder when they are cross-linked during curing. The curing process improves protein wet ability and exposes a variety of side-chain functional groups such as amines, carboxylic acids, phenols, and thiols [25]. It has also been found that adhesive strength increases due to the relaxation of internal stress and the irreversible aggregation through formation of disulfide bonds and helix-coils that occurs during curing [26,27,28]. Adsorption of protein based adhesives into wood promoted by secondary forces, including van der Waals and hydrogen bonds appears to be the predominant mechanism that has been studied earlier [29]. These animal protein adhesives have found also many other applications, such as bookbinding, paper manufacturing, gummed tape, cork composition, match heads and in sand paper manufacturing to bind the silicon, aluminum oxide emery and abrasive rings [10, 21, 22]. Enhancing the adhesive performance can be achieved by forming blends of soy protein with other natural (proteins) or synthetic resins adhesives. These formulated blends have promoted the good properties from both components to get new useful adhesive material [1,30]. This behaviour of the protein due the formulation of prepared adhesive and selection of starch for the formulation. Previous studies have show that carbohydrates in the form of oligomers, monomeric sugars and polysaccharides (from plants sources, microorganisms and exoskeletons of marine animals), have been used as wood adhesives for many years [31]. They are plentiful, low-cost, easy to apply and they can be used in wood adhesives formulations in many ways [31]. As modifiers for more expensive synthetic adhesives have characteristics such as PF and UF resins i.e. as thickeners, colloidal stabilizers

and flow controllers. Starch is one of the most abundant natural polymers [32]. Starch has been used as an adhesive in a wide range of products, including binders, sizing material, glues and pastes [32,33]. More recently, the development of a starch-based wood adhesive in interior applications has been described [32]. Starch yields adhesives with excellent affinity for polar materials such as cellulose. Ideally, the contact angle between the adhesive and the substrate should be small. This allows the adhesive to wet the surface and spread uniformly in a thin film. In this regard, starch based adhesives wet the polar surface of cellulose, penetrate into pores and thus form strong adhesive bonds [32].

CONCLUSIONS

The use of leather waste proteins by complexation with the starch was found effective as wood adhesive. In addition, the use of leather waste proteins is advantageous from economic point of view since it is low-cost source. After storage of prepared wood adhesive at room temperature, no significant change in the property was observed. It was found that the resulting adhesive formulations were stable at room temperature when kept in an air tight bottle. It is widely accepted that the application of adhesives (partially) derived from natural resources requires new approaches and novel technologies for the final product to meet the product specifications for a given application. The product should ideally display the typical advantages of natural materials such as lower toxicity, biodegradability, lower prices, ease of handling, abundance, and a renewable character. Moreover, to further improve the wood adhesive by modifying with other non-toxic chemicals is in progress.

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CITATION OF THIS ARTICLE

Omm-e-Hany Syed S S Beena Z, Aamir A and Asia N Wood Adhesives Derivation from Tannery Waste Protein: A Comparison with some Commercial Wood Adhesives. *Bull. Env. Pharmacol. Life Sci.*, Vol 4 [7] June 2015: 172-178