

Asian Journal of Biotechnology and Bioresource Technology

7(1): 1-9, 2021; Article no.AJB2T.66475 ISSN: 2457-0125

A Study on Surface Physicochemical Properties of Woods Treated in Emulsion of Waterborne Varnish with Sesame and Grape Seed Oil

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Authors' contributions

This work was carried out in collaboration between both authors. Authors HTS and IE designed the study, performed the analysis, wrote the protocol and the first draft of the manuscript. Author HTS managed the study. Author IE managed the literature searches and statistical analyzed. Both authors read and approved the final manuscript.

Article Information

DOI: 10.9734/AJB2T/2021/v7i130090 <u>Editor(s):</u> (1) Dr. Fernando José Cebola Lidon, Universidade Nova de Lisboa, Portugal. <u>Reviewers:</u> (1) Teresa Mabel Fonovich, National University of San Martin, Argentina. (2) Ali Shalbafan, Tarbiat Modares University, Iran. Complete Peer review History: <u>http://www.sdiarticle4.com/review-history/66475</u>

Original Research Article

Received 14 January 2021 Accepted 22 March 2021 Published 27 March 2021

ABSTRACT

Aims: An eco-friendly coating emulsion prepared with mixture of oils of sesame and grape seed and alkyd-based waterborne varnish. The prepared emulsion applied four different wood substrates (walnut, beech, cedar and fir) in order to be investigated for some selected surface properties.

Study Design: Several test methods were used to evaluate surface physicochemical properties of the coatings. The results obtained may be suggested for the selection of the best varnish-emulsion formulation for the improvement wood substrates and could provide useful evaluation of the test methods employed.

Methodology: The wood species of Beech (*Fagus sylvatica*), Walnut (*Juglans regia*), Cedar (*Cedrus liban*i) and Fir (*Abies nordmandiana*) were selected for the investigation. Commercially available alkyd based waterborne varnish was supplied ready to use form.

Both sesame oil (Sesamum indicum L.) and grape seed oil (Vitis vinifera L.) were obtained from a

company that produces them by the cold press technique commercially. Both oils were used as supplied, without additional processing. These oils were added to varnish at 10% and 20% proportions (volume/volume). The 5 µl of distilled water (surface tension of 72.6 mN /m) was applied on wood surface by a sessile droplet method to measure surface contact angles. The surface hardness and scratch resistance of the cured varnish layers on wood substrates were measured with using pencil hardness test procedure according to ASTM-D-3363 standard. Cross cut test also conducted according to EN ISO 2409 standard.

Experimental Findings: It was found that coated walnut samples show 9.8 to 13.5° higher contact angle values in all directions while other three wood samples only show marginally different values (0.1 to 4.3°). The highest contact value of 35.9° and 35.8° was found with samples of Wg20 and Wq10 which treated 20- and 10% grape seed oil proportions in varnish emulsion. The oils of sesame and grape seed typically contain various proportions of fatty acids fractions which are constituents of a carboxylic acid with a long, aliphatic tail. These groups could be created a strong bond in combination with alkyd resin on wood surface. However, the surface energy distribution show only marginally changes regardless of treatment levels and conditions. Therefore, there is not any clear advantage observed on surface wood surface energy levels with coating applications. For 10% grape seed oil/varnish emulsion conditions, the hardness of coated surfaces found to be 3H, 3H, 2H, 2H for walnut, beech, fir and cedar, respectively. At 20% grape seed oil/varnish coatings, all coating surfaces show H level range. For cross hatch experiments, marginally similar trend was observed with 10-and 20% grape seed oil/varnish and 10% sesame oil/varnish emulsion coated wood species. Moreover, it was ranked 2 for fir, ranked 3 for beech and ranked 4 for cedar wood at 20% sesame oil/varnish emulsion coatings. It is noticeable that a correlation was observed between cross cut and surface scratch resistance properties with coated surfaces.

Keywords: Waterborne varnish; fatty acids; sesame oil; grape seed oil; scratch resistance; contact angle.

1. INTRODUCTION

Besides decorative properties, numerous types of wood-based materials are useful for many special structural applications [1,2]. However, its dimensional stability is usually low and required special attention when applied to outdoor conditions [1-3]. Moreover, the main purpose of surface coating with transparent agent are the protecting appearance and color without changing the natural aesthetic properties [4,5]. Shellac, lacquer and varnish are generally preferred as surface coating agents for these purposes [6-8]. They are applied to the wood surface in liquid form and then cured physically, chemically or combination of both to create a hardened coating layers [7,8]. Due to restrictions and strict regulations of volatile organic compound emission (VOC) from conventional compounds, the chemical industry has emerged to developed more eco-friendly products. In this context, water-based varnish products offer interesting advantages as they produce low volatile chemical emission and are easy to prepare. Therefore, the use of water as solvent in coating formulations makes it easy applications with more ecological and safe conditions. On the other hand, these coatings are able to cure more easily with appropriate adhesion in wood

substrates. Although water-based surface treatment agents have many advantages over conventional solvent-based products, one of the main drawbacks is that they provide poor protective coating layer when applied at a thin single layer compare to solvent based coatings [6,8-11].

Scotch pine (Pinus sylvestris L.) and eastern beech (Fagus orientalis L.) wood samples at three different moisture levels were subjected to waterborne varnish treatments. It was reported that the highest coating hardness value was found with eastern beech with moisture content of 8% and 10%. It was suggested that wood type and moisture content were significant for surface adhesion resistance [12]. A detailed study was conducted for comparing varnish types (synthetic, polyurethane, water-based, acidcuring and cellulosic) and coating thickness on selected wood species (eastern beech, sessile oak, Scotch pine, black walnut, black poplar and linden) in terms of surface wear and abrasion resistance. It was found that both wood and varnish type which mainly impact on coating thickness, also have a significant impact on the abrasion resistance. The highest surface abrasion resistance was obtained with black walnut wood treated with an acid-curing varnish

and coated with three layers while the lowest was found with waterborne varnish applied on Scotch pine with only single coating. It was summarized the highest resistant coatings against wear was three layered acid-curing type while the lowest was single coated water-based type [13].

In the past few decades, the waterborne wood surface treatment agents (dyes, varnishes, lacquers) have received attention due to extent their effectiveness in an easiest way. In this sense, numerous studies have been conducted develop thermally, physically and chemically resistant coatings against both abiotic and biotic factors [6,14-18]. Therefore, this goal could only be possible through understanding the behavior of the formulations toward wood-emulsion matrix system. To address this challenge, oils of sesame and grape seed were added to alkydbased waterborne varnish system, creating a new type emulsion suitable for surface coatings of certain wood species. The certain surface properties of emulsion coatings applied to four different wood substrates (walnut, beech, cedar and fir) were investigated. Selected test methods were used to evaluate surface properties of the coatings. The results obtained may be suggested to the selection of the best varnish-emulsion formulation for the improvement wood substrates and could provide useful evaluation of the test methods employed.

2. MATERIALS AND METHODS

The wood species of Beech (Fagus sylvatica), Walnut (Juglans regia), Cedar (Cedrus libani) and Fir (Abies nordmandiana) were selected for the investigation. These woods were acquired from the Mediterranean region in Turkey. The samples were cut in the small pieces according to TS 2470 standard from defective free sapwood parts and dried (air dried, 12%) in laboratory conditions at 20°C and 50% relative humidity before being subjected to any treatments. Commercially available alkyd-based waterborne varnish was supplied in a ready-touse form in 5 L container. The varnish was utilized as an emulsion formulation suggested in company's prospective, without the any purifications. Further information on samples preparation and experimental procedure could be found elsewhere [7].

Sesame oil (*Sesamum indicum L*.) was obtained from a company which commercially produces it by the cold press technique. It had thin liquid appearance and light yellow in color. The typical sesame oil's fatty acids composition has been given as follows; C16: 0 Palmitic: 7-12%; C18: 0 Stearic: 0.35-6%; C18: 1 Oleic: 35-50%; C18: 2 Linoleic: 35-50%; C18: 3 Linolenic: Max. 0.30-0.80%; C20: 4 Arachidonic acid: max 2.62% [19]. Grape seed oil (*Vitis vinifera L.*) was also supplied from a company which also produces grape seed oil by the press technique. It was used in liquid form and light yellow in color. The typical grape seed oil fatty acids composition has been given as follows; C16: 0 Palmitic: 9-15%; C18: 0 Stearic: 3.6-7.0%; C18: 1 Oleic: 15-29%; C18: 2 Linoleic: 48-70% [20,21].

Both oils were used as supplied, without additional processing. These oils were added to at 10% and 20% proportions varnish (volume/volume) to create a surface treatment solution and applied on wood substrates in order to investigate their impact on certain properties. Each prepared solution was applied on wood samples by simply dipping in one minute of period. After end of that procedure, the small wood samples were left to dry freely under laboratory conditions. The surface energy distribution of experimental samples was determined empirically by using a suggested sessile dropped method (contact angle) [22-24]. The small wood samples were prepared in the size of 2.0x2.0 x3.0 cm in size. The samples were taken into the conditioned room and kept at 20°C (± 3°) and 65% (± 5) relative humidity until they reached constant weight. Five experiments were made for each sample groups. The 5 µl of distilled water (surface tension of 72.6 mN/m) was applied on wood surface by a special syringe. After the water drop was placed on the surface, its angle images were measured in a total time of 30 second with 5 second intervals with using ImageJ software [22-24].

The surface coating hardness and scratch resistance of the cured varnish layers on wood substrates were measured with using pencil hardness test procedure according to ASTM-D-3363 standard. The small wood samples prepared in the size of 30x8.0 x1.5 cm. Five experiments were performed for each sample groups. In this test procedure; pencils of different hardness are moved at a 45-degree angle on a coated surface until the pencils damage/marked the coating layer. The speed of the pencil tester should be between 0.5 mm/s to 1 mm/s. It started with the softest pencil to hardest in order of 6B-5B-4B-3B-2B-B-HB-1H-2H-3H-4H-5H-6H. At the end of the process, the test area was

wiped with a soft cloth and examined using a light source and a magnifying glass. With whichever pen the varnish layer was scratched, that pen number was given as the hardness value (ASTM-D-3363). Cross cut test covered the determination of the degree of adhesion (adhesion) of paint and varnish samples with a total film thickness not exceeding 250 µm. It could be applied to metallic, wood or soft surfaces in one or multiple layers. A pattern consisting of 25 or 100 squares was created with two scratches drawn at right angles to each other with a special test apparatus according to EN ISO 2409 standard. After the scratching, the surface were separated by scratching with a thin adhesive tape and the adhesion resistance of the varnish layer on the trial wood surface by comparing with the evaluation in standard as seen in Table 1 [7].

While many combinations were utilized, some code number and abbreviations were established throughout the study given in Figures and Tables. These are; B: Beech-; C: Cedar-; F: Fir-; W: Walnut-, wood samples, S: sesame oil; G: grape seed oil. 0: control, 10 and 20 are woods treated with 10% and 20% sesame oil and grape seed oil (by volume/volume) containing alkyd based waterborne varnish emulsion.

3. RESULTS AND DISCUSSION

It has already been reported that hardwoods have significantly higher holocellulose content than coniferous woods. The highest holocellulose ratio was reported as 78.9% in beech wood followed by walnut (76.4%), fir (73.88%) and cedar (52.4%), respectively. In contrast to holocellulose, higher lignin contents were reported for cedar (35.7%) followed by fir (27.34%), walnut (29.1%) and beech (22.3%) wood species, respectively [25-29]. It is well established that hardwoods generally contain less lignin but higher carbohydrates compared to coniferous woods [25]. However, hardwoods usually contain a higher extractive substance (soluble in alcohol-benzene) than coniferous woods, as well as the complexity of their cell structures [25]. These physicochemical differences between the woods, which we briefly described here are expected to have a direct effect on wood's physical, chemical and technological processes [2,30,31]. Therefore, the chemical variations might influence the woodwater interactions to some degree in addition to affecting the physical characteristics of woods.

It has been suggested that it would be useful to know the growth characteristics of wood samples used in scientific studies to determine the behavior characteristics in water [5]. In this context, the annual ring numbers in per unit area (cm), density and Fiber Saturation Point (FSP) of the wood samples used in the study are shown in Table 2. These variables could be having some certain effects on experimental results.

Class	Explanations	Typical appearance of cross-cut test conducted samples
0	The edges of the cuts are perfectly smooth	
1	The separation of the cross cuts at intersections of the coating is not more than 5%	
2	The separation of the cross cuts at intersections of the coating are between 5% and 15%. It cannot be more than 15%.	
3	The separation of the cross cuts at intersections of the coating are between 15% and 35%. It cannot be more than 35%.	
4	The separation of the cross cuts at intersections of the coating are between 35% and 65%. It cannot be more than 65%.	
5	The separation of the cross cuts at intersections of the coating are higher than fourth class.	

Table 1. Cross cut evaluation table

Wood samples	Annual ring number per unit (Cm)	Density (g/cm ³)	FSP (%)
Walnut	2	0.79	25.59
Beech	3	0.69	31.62
Fir	5	0.46	29.01
Cedar	7	0.54	29.06

 Table 2. General characteristics of woos

The contact angle values of wood species treated with waterborne varnish emulsion prepared with oils of sesame and grape seed at two proportions (10 and 20% by volume) are shown in Table 3. Although the measurements on the radial surfaces may be sufficient, calculations were made taking into consideration also tangent surface and the average contact angle value of two surfaces of wood samples (radial+ tangent surface/2) are presented.

When the radial surfaces were examined, the control's contact angles (Θ) of 32.7°, 23.5°, 21.4° and 19.2° were measured in order of walnut, cedar, beech and fir. However, when average values are considered the highest value of 30.8° was found for walnut, followed by cedar (Θ : 21.3°), beech (Θ : 20.7°) and fir (Θ : 19.6°), respectively. Except walnut samples which show 9.8 to 13.5° higher contact angle values in all directions, other three samples only show marginally different values $(0.1 \text{ to } 4.3^{\circ})$. Moreover, small variations in all coated samples were obtained regardless of proportions and type of oil by even changing the measured surfaces. But it is noticeable that walnut results show the highest contact angle values in all conditions. The highest contact value of 35.9° and 35.8° was found with samples of Wg20 and Wg10. However, these values are only a few points different than control (W0; Θ : 32.7°). It is noticeable that it was very difficult to put a precise ranking for other samples. It has already been reported that water contact angle properties are closely related to surface energy and hydrophilic/hydrophilic properties [7]. If the water contact angle is less than 90°, the substrate surface is considered to be hydrophilic, or conversely, if the water contact angle is greater than 90°, the solid surface is considered to be hydrophobic. In our study, it was clearly seen that selected oil substances and combinations with waterborne varnish have not considerably impact on hydrophobic properties on wood species.

It could be assumed that the chemical constituents of prepared emulsion typically contain various proportions of alkyd resin and

fatty acids fractions (i.e., oleic, linoleic, linolenic, palmitic, stearic, etc.). The chemical formulas of the most common oleic, linoleic and linolenic acid are shown in Fig. 1. However, the fatty acids in both oils typically consist on a carboxylic acid group with a long, aliphatic tail. It is a wellestablished property for fatty acids that the hydrocarbon chain is highly non-polar and therefore water insoluble (hydrophobic). Hence the carboxyl end dissolves and typically forms a layer with water (hydrophilic end) while the hydrocarbon tale remains outside the water surface (26). Because forming new bonds on the wood surface is a key for creating hydrophobic properties, therefore a certain type and number of bonds within the wood must be formed to create resistance against water (Fig. 1). Due to existence of OH groups in wood structure, the hydrogen bonds generally have bonding strength between 15-25 ki/mole is primarily established in a liquid-wood interaction. On the other hand, the covalent bond energies are range of 250-600 kj/mole, which are very stable and could not be affected by water. In our study, the surface contact angle values show only marginally changes regardless of treatment levels and conditions. Therefore, there is not any clear advantage observed against wood surface energy level reduction with coatings. It could be hypothesized that the combination of varnish emulsion-wood could not considerably reduce surface energy, due to the not formation a appropriate strong bond. As a result of that, the treatment does is not promote the increasing contact angle of the wood species. The experimental results and literature information clearly support this hypothesis.

The changes in pencil hardness degrees of wood are comparatively shown in Table 4. In general, it can be observed that the surface hardness of the wood samples was decreased (softer) in all coatings regardless of proportions of vanish emulsion. However, at 10% grape seed oil/varnish emulsion conditions, the hardness found to be 3H, 3H, 2H, 2H for walnut, beech, fir and cedar, respectively. At 20% grape seed oil/varnish coatings, all wood's hardness was found to be in H level range. Hardness of 3H was found for walnut and fir while it was 2H for cedar and beech samples. Moreover, at 10% sesame oil/varnish coatings, the hardness found to be B level in all samples. At 20% sesame oil/varnish coatings, it was determined 3H for walnut and fir while 2H for cedar and beech samples. It could be concluded that modification of wood surface hardness with changing emulsion formulations is important in terms of providing different scratch resistance of surfaces.

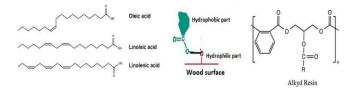


Fig. 1. Wood surface bonding potentials with fatty acids	and alkyd resin
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Wood samples	Radial surface	Tangent surface	Average
B0	21.4	19.9	20.7
CO	23.5	19.1	21.3
F0	19.2	20.0	19.6
W0	32.7	28.9	30.8
Bg10	21.6	21.7	21.7
Cg10	22.5	21.8	22.2
Fg10	21.3	24.2	22.8
Wg10	35.8	24.8	30.3
Bg20	20.7	20.1	20.4
Cg20	23.1	22.1	22.6
Fg20	23.4	20.9	22.2
Wg20	35.9	22.2	29.1
Bs10	20.6	18.9	19.8
Cs10	23.0	19.1	21.1
Fs10	23.9	21.1	22.5
Wg10	29.3	25.1	27.2
Bs20	23.1	20.4	21.8
Cs20	24.3	24.4	24.4
Fs20	21.1	19.6	20.4
Ws20	25.3	24.8	25.1

Table 3. Surface contact	angle values (O; o	degree) of wood species

Table 4. Coated surface hardness properties (ASTM D-3363) of wood species

Woods	Hardness
Bg10	3H
Cg10	2H
Fg10	2H
Wg10	3H
Bg20	2H
Cg20	2H
Fg20	3H
Wg20	3H
Bs10	В
Cs10	В
Fs10	В
Ws10	В
Bs20	3H
Cs20	В
Fs20	2H
Ws20	3H

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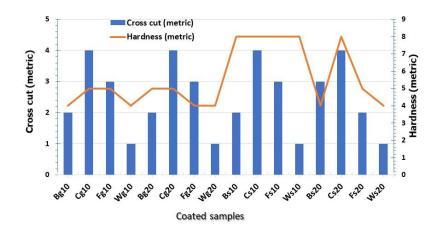


Fig. 2. Cross cut and hardness changes of wood species

The cross-cut test is a visual comparison method as compared in Table 1 for a thin coating layer (paint, varnish, film) up to 250µm. The coating is cut into small squares, thereby reducing lateral bonding, and the coating adhesion is assessed against ASTM D 3359 standard. Fig. 2 show cross cut properties (as given ranks in metric) values and hardness (hardness values calculated as in metric) of wood species coated with oils of sesame and grape seed at 10 and 20% proportions comparatively. In order to compare the two properties, cross cut and surface scratch resistance (pencil hardness), a practical calculation was designed through establishing pencil hardness values in a numbers and letters scale from hardest surface to softest one in the order that follows; 6B: 13 (the softest one); 5B: 12; 4B:11; 3B:10; 2B;9; B: 8; HB: 7; H: 6; 2H:5; 3H:4; 4H: 3; 5H: 2; 6H: 1 (the hardest one). In general, more or less similar trends in cross cut properties were observed with 10-and 20% grape seed oil/varnish and 10% sesame oil/varnish emulsion coatings. However, at 20% sesame oil/varnish coated samples show a variable cross cut property that it was ranked 2 for fir, 3 for beech and 4 for cedar wood. Although some small variables could be observed with beech and fir woods which were coated with at 20% sesame oil/varnish emulsion. walnut show the highest adhesion (ranked 1) whereas cedar show the lowest adhesion (ranked 4), regardless of treatment conditions emulsion proportions. It could and be summarized that hardwood (walnut and beech) samples show better adhesion properties than softwood species (fir and cedar) with coatings in certain oils of sesame and grape seed mixed with waterborne varnish emulsions. When Fig. 2

is carefully analyzed, it might be seen a sort of correlation between these two parameters.

4. CONCLUSION

The two-component solvent-based (synthetic varnishes) are commonly utilized as surface protective agents in many applications. However, these kinds of treatments are typically causing volatile organic compound (VOC) emissions to the atmosphere. Therefore, it is needed to utilize more environmentally friendly coating agents in woodworking industry. In this context, many surface coating agents with different formulations have emerged in the recent years. As a result of these studies, water-based varnish systems containing very low levels of volatile organic compounds have been prepared from various polymers, and their popularity have increased gradually. But these transparent finishes are applied in very thin layers and their effectiveness is limited. For that reason, in order to improve surface coating properties, oils of sesame and grape seed were added to these water-based varnish systems, for making a special formulation for certain wood species. It was observed that specially prepared mixture of varnish emulsion may be an alternative surface agent to helps in maintaining the certain wood properties that could be easy to prepare, apply and cleaned quite easily. But the effectiveness of these coating emulsion may be extended with multiple coatings and/or applied in special wood elements.

ACKNOWLEDGEMENTS

The authors wish to thank Isparta University of Applied Sciences, Scientific Research

Coordination Division (ISUBU-BAP) for financial support and contribution to this research. This study was carried out within the ISUBU-BAP project no: 2019-YL1-0040.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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> Peer-review history: The peer review history for this paper can be accessed here: http://www.sdiarticle4.com/review-history/66475