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FACILITATING THE APPLICATION OF STARCH PASTE IN THE CONSERVATION OF PAPER ARTWORKS

Kobra DADMOHAMADI [1*]

[1] Department of Calligraphy and Persian Miniature, Alzahra University, P.O. Code: 1993893973, Tehran, Iran

Abstract

Starch paste is an adhesive that is used in the field of book and paper conservation. This adhesive has a very short shelf life, and for this reason, it must be prepared in small quantities. Also, preparing starch paste for use in protection is a time-consuming process. This research was done with the aim of facilitating the use of starch paste in the conservation of paper works, long-term storage of starch pastes without fungicidal additives and increasing its durability in the long term without changing the quality of the adhesive. The application of fresh starch pastes and activated starch film increased the pH of the samples coated by these adhesives compared to the control samples without adhesive. The samples covered by the activated starch film after moist-heat aging and light aging had the highest pH compared to other samples. The samples coated with fresh starch paste showed the lowest amount of color changes (ΔE) after moist-heat aging and light aging, respectively. Application of fresh starch paste and activated starch film increased the tensile strength of the samples. After aging, the tensile strength of the samples decreased compared to the previous stage, but the tensile strength of the samples covered by these adhesives increased compared to the control samples without adhesives. The adhesion of the samples decreased after aging. The samples coated with fresh starch paste had the highest adhesion after light aging. Also, after moist-heat aging, the samples covered by the activated starch film had the highest amount of adhesion.*

Keywords*: Rice starch paste; Starch film; Activation; Paper conservation; Adhesive*

Introduction

Adhesives are among the materials that are widely used in the conservation and restoration of paper. Adhesives are materials that are used to stick or connect between two surfaces and exist in the form of liquid, paste, powder or dry layer [1]. Adhesives are used in paper conservation for various purposes, such as repair of tears and gaps, consolidation, fixation of soluble inks, sizing, or lamination. In order to be used in cultural heritage conservation, adhesives need specific qualities, such as a compatible pH; chemical inertia with the substrate; a long period of use; colour stability over time; reversibility; and low bioreceptivity [2]. One of the most important adhesives used in the restoration of paper works is starch paste. This paste is still widely used in restoration [3]. Starch paste, the most used adhesive in paper conservation is a natural polymer composed of two types of molecules: amylose (linear glucose chains) and amylopectin (ramified glucose chains) [4].

Since the advent of papermaking, starch has been used as an important additive in paper making [5]. Starch does not dissolve until it reaches 55-80 degrees centigrade. At this temperature, the seeds become soft and start to wane. This causes a rapid increase in viscosity and then its decrease and the complete wane of the seeds. As a result, a semi-transparent white solution is obtained [6]. Due to its polymeric nature, starch has the ability to form a film. In

* Corresponding author: k.dadmohamadi@yahoo.com

addition, due to its cheap price and abundance, much attention is paid to it [7]. Starch as a raw material in the production of adhesives has many advantages, including renewability, biodegradability, abundance and stability in price [8].

Examining the traditional starch paste used by paper art restorers has shown that starch paste has been used since ancient times and if this paste is made from wheat starch granules and not cooked flour, it will be reversible. Generally, the removal of starch paste depends on the quality of the starch used and the method of making the paste. Restorers of works of art on paper are more inclined to use traditional adhesives that have been tried and tested over time because they can control the process of destruction of these adhesives [9]. Starch paste is exposed to relatively fast biological attack within a few days after preparation. Addition of fungicide can reduce the degradation process to some extent. But it is recommended that in order to avoid reducing the amount of adhesion, the paste should be made fresh weekly. Because fungicides cause yellowing of paper over time, some restorers do not use any kind of fungicide for starch paste, but they make new paste regularly. Some also prefer to store the paste in the refrigerator.

However, the adhesive should not be stored at the low temperature of a home refrigerator (39.2 F° or 4C°), because it turns it into grains and the quality of the paste is lost [3]. Analysis of chemical stability and fungal bioreceptivity of five adhesives currently used in the paper conservation field (starch paste, unsupported Archibond, carboxymethylcellulose, hydroxypropylcellulose and methylcellulose) has shown that Starch paste was the most bioreceptive adhesive, but on other hand was also the most stable adhesive to artificial ageing, regarding colour alteration, degree of polymerization and pH [10]. The use of kudzu starch as an adhesive in the restoration of cultural heritage has shown that kudzu had an optimal chromatic behaviour during accelerated aging process compared to common starch adhesives. On the other hand, the daidzein, a natural antimicrobial compound implicit in Kudzu starch, confirmed the resistance to microorganisms in this preliminary approach. The evaluation of the adhesive capacity, and the reversibility of the starches, suggest that Kudzu starch is a valid adhesive in the field of paper restoration.

Thus, the potential of this starch in the conservation of Cultural Heritage is evidenced and its use as cleaner, resistance to biological colonization and consolidant is promising [11]. Analysis of the preparation of wheat starch paste after collecting over 50 recipes from publications and conservation professionals, has shown that parameters of starch source, pre-soaking, starch-to-water ratio, cooking method, cooking duration, cooking temperature, sieving and storage time/method affect the quality of the end-product for conservation use [12].

Therefore, in the usual methods of preparing starch paste, the paste must be used immediately and usually the remaining paste is thrown away or if it is kept, there is a possibility of fungus and mold growing on it. It is also difficult to maintain and its shelf life is very short. In this research, the starch paste used in the restoration of paper works is prepared in the form of a film and then at the time of use, the starch film is activated by water and is used. In this method, the durability of the adhesive increases and there is no need to use fungicide. It will also be easier to use. In addition, it will be economical. Starch film can be stored much longer than paste stored in the refrigerator or with fungicides. It is readily available and easily portable if work is to be done outside the laboratory. In this research, the characteristics of activated starch film and fresh starch paste were compared.

Materials and Methods

Materials

Preparation of rice starch

In this research, rice starch was prepared according to the standard number 3-381 of the National Standard Organization of Iran. First, 100 grams of rice were washed with water and soaked in water for 24 hours to soften, then the water was separated and the rice was pounded in a mortar. In the next step, pounded rice was mixed with 500 ml of water and placed on the heater. After thickening the water containing rice, the obtained solution was separated with a strainer and used as a paste for sampling. The starch solution was mixed with water at different

ratios and finally, a ratio of 3 to 1 (3 parts paste and 1 part water) due to its suitable concentration, it was used as a paste for sampling.

Preparation of starch film and its activation

Standard number 4797 of the National Standard Organization of Iran was used to prepare the starch film. First, the obtained starch and water were combined with each other at a ratio of 1:1, then the prepared solution was poured on a Melinex polyester sheet on a flat surface and after drying, it formed a thin layer of starch film (Fig. 1). In the next step, in order to prepare the samples, the starch film was reactivated, in such a way that some of this film was placed in a container and then some hot water (10 grams of starch film + 30mL of water) was added to it and the container was placed in a bain marie bath (water bath) at 60°C for 10 minutes. After the starch was prepared, it was used as an adhesive.

Fig. 1. Preparation of starch film

Preparation of samples

Tissue paper (Japanese) 9.10g/m^2, GIFU was used to prepare the samples for the desired tests. This paper has a neutral pH and is less thick than other types of tissue paper and it is usually used for restoration and lining of paper works. The prepared starch pastes (fresh starch paste and activated starch film) were gently placed on the tissue paper by a brush, and the papers were covered with the prepared pastes. Then they were dried at ambient temperature. After that, the desired tests were performed on the samples before and after aging. For ease of operation and the least possible error, the samples were coded according to Table 1.

Table 2. Prepared samples and their abbreviated code

Sample code	Description of treatment
P	control paper without starch paste
PS	Paper coated with fresh starch paste
PSF	Paper coated with activated starch film
SF	Starch film

Methods

Accelerated aging methods

Accelerated aging was used to investigate the changes in the samples during the aging process. The investigated changes included pH changes, color changes, tensile strength, FTIR-ATR spectroscopy and adhesion resistance of the samples. This test was performed by two moist-heat aging methods according to ASTM standard D7414-96 at a temperature of 90C° and a relative humidity of 50% for 384 hours. This test was performed using a Memmert oven with a maximum temperature of 120C° and 600 watts and 220 volts [13]. In light aging according to ASTM standard D6789-02, the samples were exposed to OSKAM xenon lamp HQI-BT400

made in Slovakia for 360 hours. The radiation angle was 45 degrees and the temperature on the surface of the samples was between 20 and 30C°, by changing the distance of the lamp from the samples [14]. After the end of the considered time, the changes made in the samples were compared with those before aging.

pH determination

pH changes in the samples were measured before and after aging according to ISO 6588-1 (cold extraction method) and by Metrohm 744 digital pH meter [15].

Colorimetry

The color changes of the samples were examined before and after aging using a color tecto alpha hand-held colorimeter by Salutron Messtechnik, according to the CIE system using ISO 11644-4. In the CIELAB colorimetry method, the values of L* (light-dark), a* (red green), and b* (yellow-blue) are shown [16]. To evaluate the changes of these factors in the samples, the following equations were used (Eq. 1).

$$\Delta E_{Lab} = \sqrt{(L^*_2 - L^*_1)^2 + (a^*_2 - a^*_1)^2 + (b^*_2 - b^*_1)^2} \quad [1]$$

In these equations, L^*_1, a^*_1 and b^*_1 demonstrate samples without agingand L^*_2, a^*_2 and b^*_2 demonstrate those after the aging process, also ΔL^*, Δa^*, Δb^* and ΔE^* shows the total changes of colors in the CIElab.

ATR-FTIR spectroscopy

The structural changes that occurred in the samples before and after aging, as well as the comparison between the samples containing starch paste and the control samples with each other by non-destructive testing using infrared spectroscopy with attenuated total reflection (ATR-FTIR) was investigated. In this method, each of the samples was placed under the sensor of the Nicolet-nexus 470 infrared spectrometer and this spectrometry was performed by the surface method (ATR) of the samples.

Tensile strength measurement

The tensile strength of the samples was measured before and after aging according to ISO 1924-3 [17]. To measure the tensile strength, the paper was cut to 150 by 15mm and placed vertically between the upper and lower jaws of the machine, which was 100mm of the paper length between the two jaws, and the tensile force was applied to it. When the paper strip is torn in half, the force specified by the machine is the maximum tensile force that the paper has withstood to the point of rupture. The amount of tensile strength of the samples was obtained according to Formula 2, which is provided in the standard (Eq. 2).

$$\sigma_T^b = \frac{\overline{F}_T}{b} \quad [2]$$

where: σ_T^b is tensile strength in (kN/m) unit, $\overline{F}_T$ is the average of maximum tensile force in N unit, and b is the width of sample in mm unit.

Adhesion resistance

Determining the adhesion resistance of the samples and examining the changes in their adhesion before and after aging were done according to the ASTM D1876 standard [18]. The test plates were prepared with a width of 25mm and a length of 305 mm and 241mm of their length were connected to each other using a cold press machine. This test was done using INSTRON 5566-H1730 tensile strength device. During the test according to the standard, the force diagram was recorded according to the peeled length and after the test, the obtained results were determined based on the peel resistance of at least 127mm of the connection line after the first peak of the graph (Fig. 2).

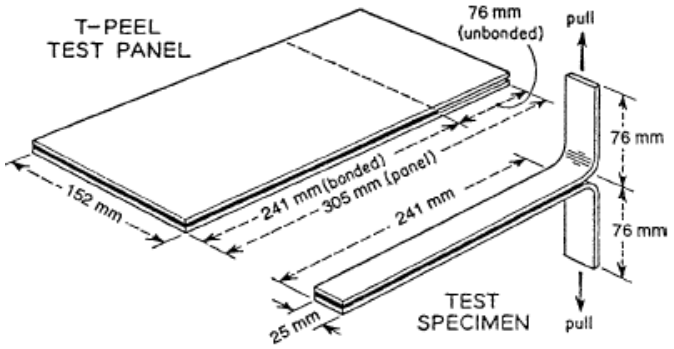

Fig. 2. Schematic design of the samples prepared for the adhesion resistance test

Results and Discussions

pH changes

The application of fresh starch paste and activated starch film increased the pH of the samples coated by these adhesives compared to the control samples without adhesive. In the analysis of the acidity of the samples after two stages of aging, it was found that the pH of none of the samples was lower than 6. The pH of most of the samples remained in the range of 6 and 7 after aging. But on average, the highest level of acidity after two stages of aging corresponds to the PS sample (paper coated with fresh starch paste) with a pH of 6.1 after light aging and 6.01 after moist-heat aging. The lowest level of acidity also corresponds to SF (starch film) samples with pH 8.35 after light aging and pH 8.20 after moist-heat aging (Fig. 3). Conducting hydrolysis and oxidation reactions leads to the breaking of the cellulose chain and results in the phenomenon of paper acidification. In these conditions, the pH of the paper is reduced, its color tends to yellow, and it becomes very fragile and loses its mechanical strength [19]. Hydrolysis is the most important chemical reaction that breaks down cellulose molecules. In this reaction, the chain is broken by adding a water molecule to the structure. This reaction mostly occurs in the amorphous parts of the cellulose chain. Oxidation is another reaction that leads to the breaking of cellulose chains. In cellulose, further oxidation reactions cause the formation of carbonyl groups from the hydroxyl groups of glucose units. Then, with the continued oxidation of carbonyls, carboxylic acids are formed [20]. Therefore, the pH of paper can be considered as one of the indicators for cellulose degradation.

Color changes

To study the amount of color changes of the samples before and after aging, the average change of L*, b* factors of each sample was calculated. In figure 4, the changes of the L* factor (lightness-darkness) of different samples compared to the control samples are shown. Based on the obtained results, before aging, the amount of L* factor in PS (paper coated with starch paste) and PSF (paper coated with activated starch paste) samples has decreased compared to the P sample (paper without paste), that this reduction rate was higher in the PSF sample, Based on this, it can be concluded that the use of starch paste and activated starch paste on paper has darkened the color of the paper.

In the control sample, the most color changes occurred after moist-heat aging, and the samples became a little darker after aging, but this change was very slight. In the PS sample, the most color changes occurred after light aging. The color changes created in these samples were also very slight. In the PSF sample, the L* factor has been greatly reduced after light aging, and as a result, the color of the samples has become darker. After moist-heat aging, the L* factor also decreased, but its amount was very low. In the SF (starch film) sample, the L* factor after

light aging has decreased to a greater extent than moist-heat aging, and the color of the sample has become darker after light aging, while after moist-heat aging, less color changes have taken place in it.

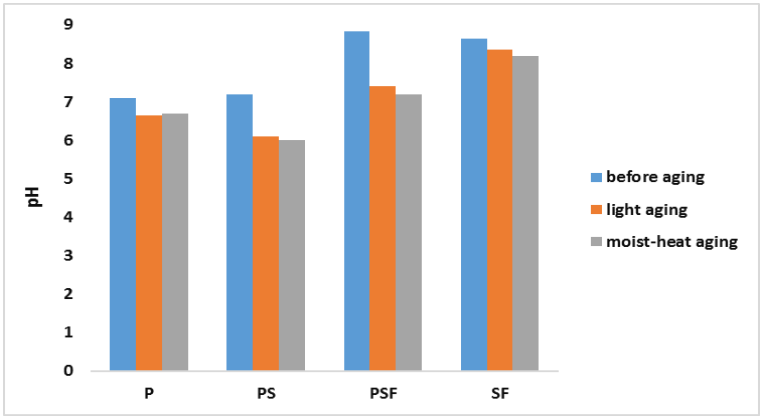

Fig. 3. The pH of the samples before and after aging

Accelerated aging has caused a decrease in the L* factor and as a result a slight decrease in the brightness of the samples. Many products of the paper aging process, such as oxidation products, cause the paper to darken. As the paper gets older, its color changes and darkness increases [21]. The yellowing of paper materials and the reduction of its brightness in aging occur as a result of the decomposition of paper due to accelerated aging. So that aging causes the oxidation of cellulose and the formation of carbonyl chromophores [22].

What is very important in examining the color changes created in the samples is the changes in factor b* (yellow blue). In sample P after aging, b* factor has increased, and this increase after moist-heat aging is more than light aging, and the color of the sample is yellow. In the PS sample, after light aging, the factor b* decreased and the color of the sample became lighter, but after moist-heat aging, the amount of this factor increased and the color of the sample became darker. In the PSF sample, the color of the samples has darkened after both stages of aging, but more color changes have occurred in the samples after light aging. In the SF sample after light aging, factor b* increased and the color of the samples turned yellow, but after moist-heat aging, factor b has decreased slightly (Figs. 4-6).

In addition to the separate analysis and investigation of each of the L*, b* factors in the samples, the total color changes (ΔE*) of the samples were also investigated. For this purpose, the samples before aging were compared with the samples after moist-heat aging and light aging to determine the total color changes. The total color change (ΔE*) of the samples is shown in figure 6. Comparing the total color changes (ΔE*) of the samples before aging with the samples after moist-heat aging and light aging, shows that, in general, the index of color changes in the samples has increased over time. PSF and SF samples have the highest amount of color changes after aging. The lowest amount of color changes after light aging is related to P sample and after moist-heat aging, it is related to PS sample.

The index of color changes in PS, PSF and SF samples after moist-heat aging has decreased compared to light aging, but it has increased in P sample. The comparison of the color changes in the samples shows that more color changes have occurred in the PSF sample after light aging and moist-heat aging than the PS sample. This is caused by the chemical changes created in the paste and paper during the aging process. These changes can accelerate the oxidation and hydrolysis in cellulose and hemicellulose and increase the light-absorbing chromophores and carbonyl group and form yellow chromophores and increase the color change on the paper surface [23].

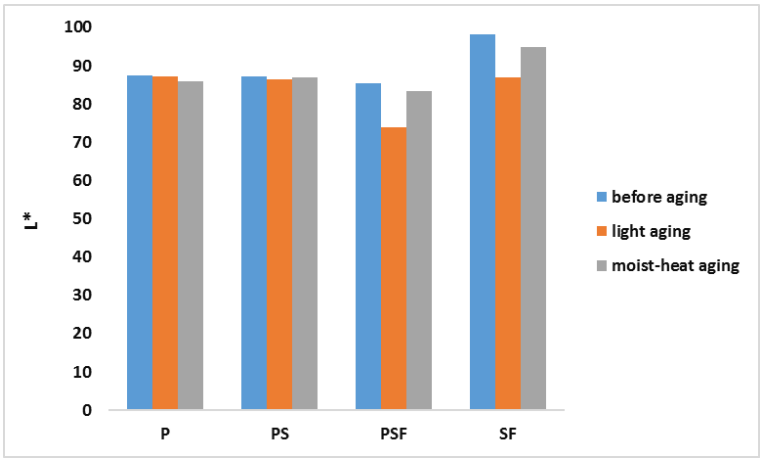

Fig. 4. Changes in the L* factor (light-dark) of the samples

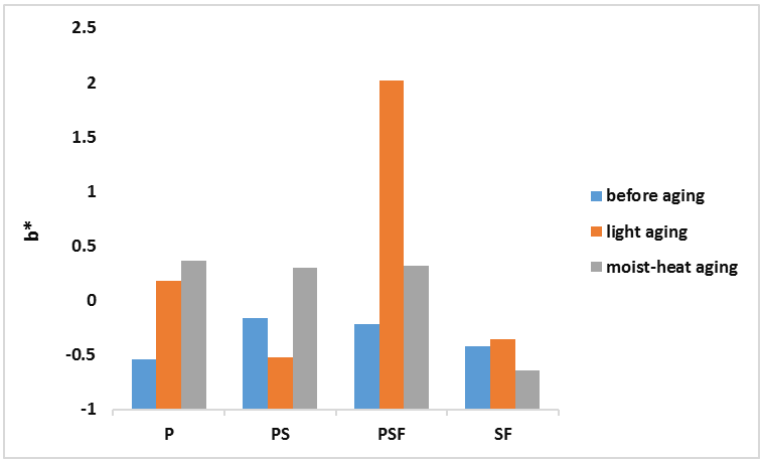

Fig. 5. Factor b* changes (yellow-blue) of the samples

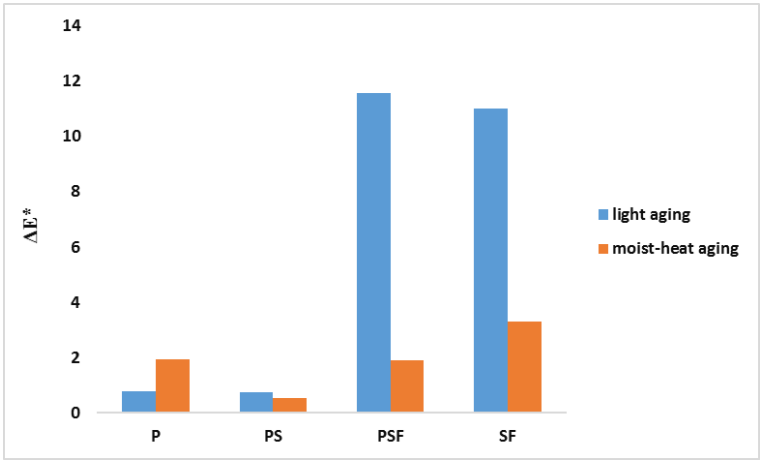

Fig. 6. The total color changes (ΔE*) in the samples

ATR-FTIR spectroscopy

The change in absorption intensity in some areas, such as the increase in absorption in the range of 1670-11680cm^{-1}, that is the C=O double bond region, which is related to carbonyl groups, In PS and PS samples after moist-heat aging and light aging, it can be caused by the oxidation of these double bonds and causes the samples to change color [24]. The appearance of a peak in the range of 1100cm^{-1} in PS, SF samples and in PSF samples, after moist-heat aging, indicates the tensional vibration of C-O in cellulose.

Changes in this absorption indicate the breaking of the oxygen bridge in the polymer chain of polysaccharides. The increase in absorption in the range of 1500-1400cm^{-1} due to the asymmetric bending vibration of the CH group indicates the increase of this group, which confirms the transverse destruction [25]. In general, the increase in the intensity of peaks in moist-heat aging was more than in light aging and the structural changes created in the samples due to moist-heat aging were more than light aging (Figs. 7 - 9).

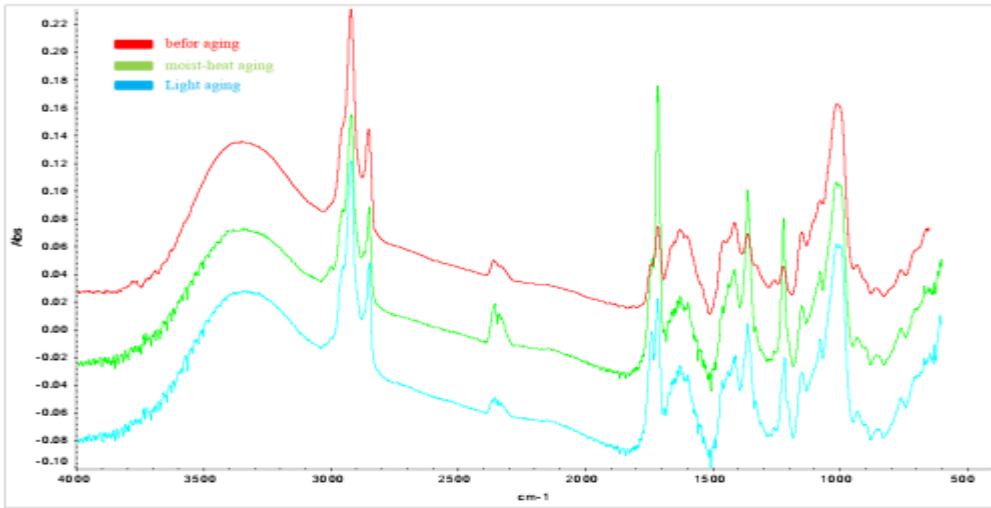

Fig. 7. ATR-FTIR spectrum of PS sample, paper coated by starch paste before and after aging

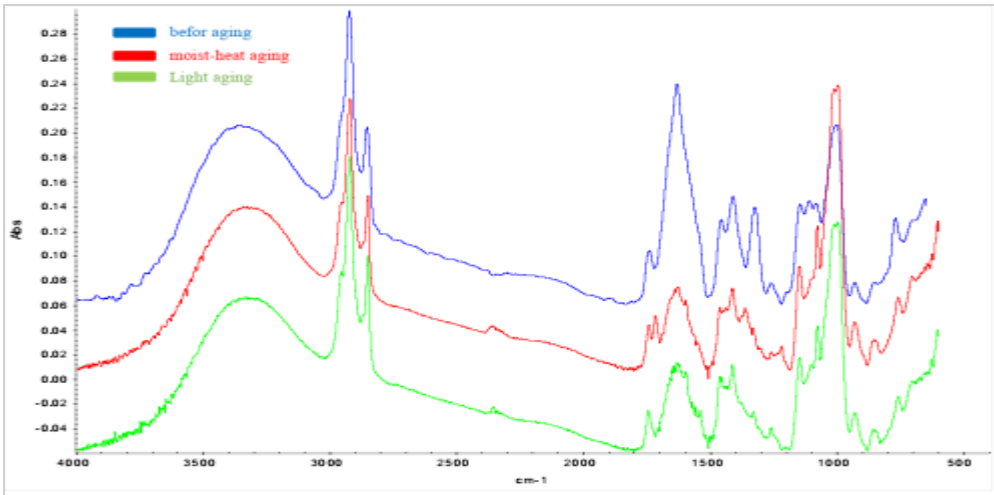

Fig. 8. ATR-FTIR spectrum of SF sample, before and after aging

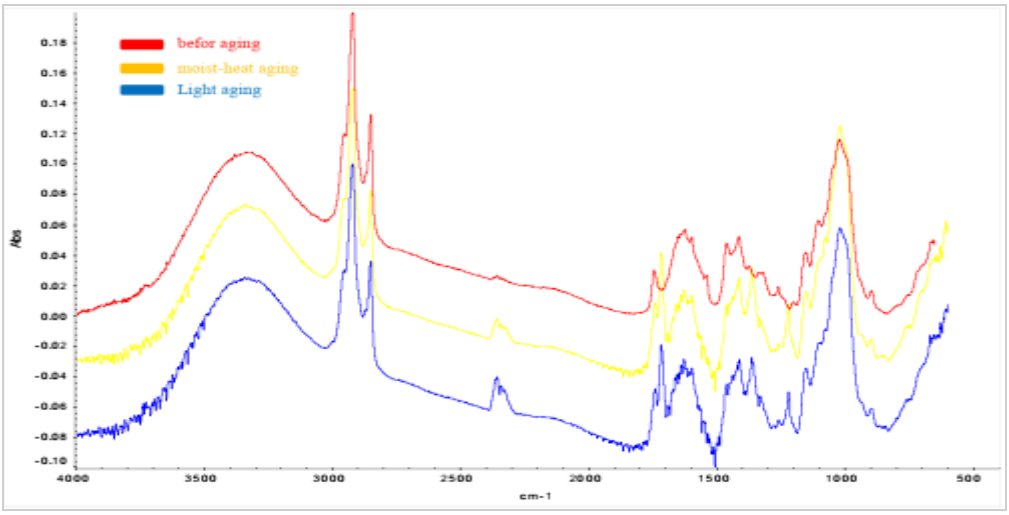

Fig. 9. ATR-FTIR spectrum of PSF sample, paper coated with activated starch film before and after aging

Tensile strength

The results obtained from the tensile strength test of the samples show that the amount of tensile strength of the samples covered by starch pastes has increased compared to the control samples without paste before aging. This increase in tensile strength and rupture is due to the placement of the starch paste used as a layer on the paper, which increases the strength. Due to the use of starch paste, the surface area of contact between the fibers and the material has been developed, which has improved fiber-polymer adhesion, better stress transfer to the starch paste, and improved tensile mechanical properties. In general, the starch paste caused better interaction and mixing of the starch paste and paper fibers, and this increased the strength of the samples [26].

The highest tensile strength before aging is related to the PS sample. The amount of tensile strength of PSP samples has decreased compared to PS samples. After light aging and moist-heat aging, the highest amount of tensile strength is related to the PS sample, and the tensile strength of the PSF sample is reduced compared to it (Fig. 10).

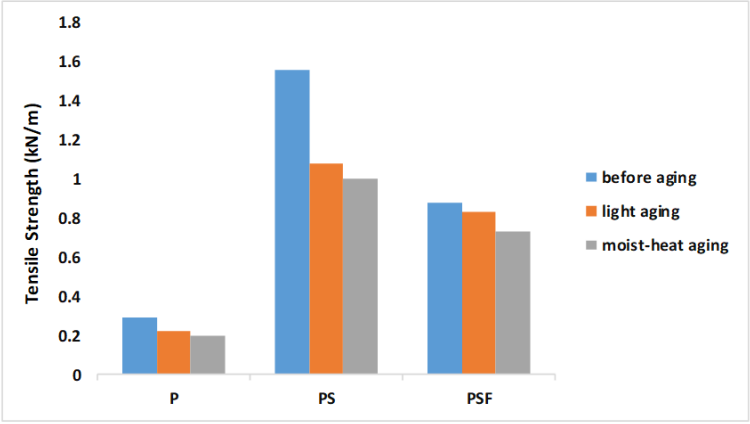

Fig. 10. Tensile strength of samples before and after aging

Examining the percentage changes in the tensile strength of the samples after light aging and moist-heat aging shows that tensile strength of the PSF sample increased by 20.54% after light aging compared to before aging and by 13.69% after moist-heat aging. The tensile strength of other samples has decreased compared to before aging. After light aging and heat-moisture aging, the lowest tensile strength reduction is related to P sample with 24.13% and the highest tensile strength reduction is related to PS sample with 30.76%.

Adhesion resistance

As seen in figure 11, the results obtained from the adhesion resistance test show that the adhesion of the samples coated with fresh starch paste and activated starch film after light aging and moist-heat aging has decreased. Unlike the PSF sample, the adhesion resistance of the PS sample decreased more after moist-heat aging compared to light aging. The highest amount of adhesion before aging is related to the PS sample. After light aging, the highest amount of adhesion resistance is related to the PS sample and the lowest amount of adhesion resistance is related to the PSF sample. After moist-heat aging, the highest amount of adhesion resistance is related to the PSF sample and the lowest amount is related to the PS sample.

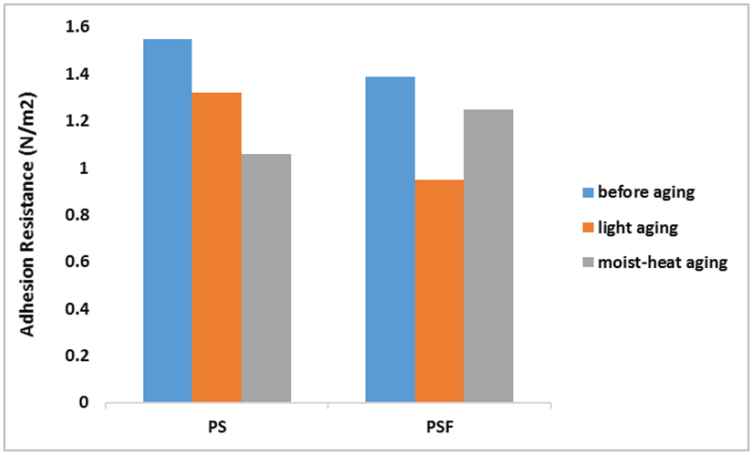

Fig. 11. Changes in adhesion resistance of samples before and after aging

The aging process has an effect on the adhesion resistance of the samples, because after two stages of aging, the adhesion resistance of the samples has decreased. In conditions of accelerated aging, hydrolysis and oxidation occur faster, this leads to a decrease in adhesion resistance. Placing the papers covered by starch paste in conditions of accelerated aging cause's hydrolysis and thus increases the speed of the degradation process. Because with increasing temperature, the speed of molecules also increases and the concentration of hydrogen ions increases, based on this, the destruction reaction increases [27].

Conclusions

The results of the colorimetry of the samples before and after aging show that all the samples had some color changes, among them, the highest color changes were related to PSF (paper covered with activated starch film) and SF (starch film) samples, and the lowest color changes were related to P sample (control sample without paste). Measuring the acidity of the samples also showed that after aging, the acidity of the samples decreased. The lowest level of acidity after two stages of aging is related to the PS sample, whose pH has decreased from 7.2 before aging to 6.1 after light aging and 6.01 after moist-heat aging.

The highest level of acidity also corresponds to the SF samples, whose pH has decreased from 8.63 before aging to 8.35 after light aging and pH 8.20 after moist-heat aging. The comparison of the results of the tensile strength test of the samples shows that the highest tensile strength in the stage before aging is related to the PS sample. After light aging and moist-heat aging, the highest amount of tensile strength is related to the PS sample, and the tensile strength of the PSF sample has decreased compared to it. Examining the results of measuring the adhesion resistance of the samples also shows that the highest amount of adhesion in the stage before aging is related to the PS sample.

After light aging, the highest amount of adhesion resistance is related to the PS sample and the lowest amount of adhesion is related to the PSF sample. After moist-heat aging, the highest amount of adhesion resistance is related to the PSF sample and the lowest amount is related to the PS sample.

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