EVALUATION OF THE FUNCTION OF ADHESIVE TISSUE PAPER MADE WITH STARCH PASTE FOR PAPER CONSERVATION AND RESTORATION

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Date Received: 12/07/2023

Date Revised: 24/07/2023

Date Accepted: 31/07/2023

ABSTRACT

The purpose of this research is to facilitate the use of starch paste in the restoration of paper works. Especially the works where ink and colors used are not sensitive to water. In this research, starch paste with a ratio of 3 to 1 (3 parts paste and 1 part water) was used. The use of starch paste increased the pH of the samples coated with this paste compared to the control samples without paste. The samples coated with starch paste had the lowest pH after moist-heat aging compared to other samples. The samples coated with starch paste increased the lowest amount of color changes (ΔE) after moist-heat aging and light aging, respectively. The use of starch paste increased the tensile strength of the samples. After light aging and moist heat aging, the tensile strength of the samples decreased compared to the previous stage, but the tensile strength of the samples coated with starch paste increased compared to the control samples without paste. The results of the adhesion resistance test of the samples showed that the amount of adhesion of the samples decreased to a greater extent after moist-heat aging compared to light aging.

Keywords: Starch Paste, Adhesive Tissue Paper, Adhesive, Paper works, Conservation and Restoration.

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 two surfaces and exist in the form of a liquid, paste, powder, or dry layer (Prajapati, 2005). 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, color stability over time, reversibility, and low bioreceptivity (Zervos & Alexopoulou, 2015). One



of the most important adhesives used in the restoration of paper works is starch paste. This paste is still widely used in restoration (Horie, 2010). Starch paste, the most commonly used adhesive in paper conservation, is a natural polymer composed of two types of molecules: amylose (linear glucose chains) and amylopectin (ramified glucose chains) (Alexopoulou & Zervos, 2016). Since the advent of papermaking, starch has been used as an important additive in papermaking (Vodopivec et al., 2004). Starch does not dissolve until it reaches 55–80 degrees Celsius. At this temperature, the seeds become soft and start to wane. This causes a rapid increase in viscosity, then a decrease and the complete waning of the seeds. As a result, a semi-transparent white solution is obtained (Daniels, 1998). Due to its polymeric nature, starch has the ability to form a film. In addition, due to its cheap price and abundance, much attention is paid to it (BeMiller & Whistler, 2009).

Starch as a raw material in the production of adhesives has many advantages, including renewability, biodegradability, abundance, and stability in price (Agboola et al., 1990). 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 destruction process of these adhesives (Fairbrass, 1995). Starch pastes are exposed to a relatively fast biological attack within a few days after preparation. The 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 be made fresh weekly. Because fungicides cause the paper to yellow 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 4 °C) because it turns into grains and the guality of the paste is lost (Horie, 2010).

Analysis of the chemical stability and fungal bioreceptivity of five adhesives currently used in the paper conservation field (starch paste, unsupported Archibond, carboxy methyl cellulose, hydroxy propyl cellulose, and methyl cellulose) has shown that starch paste is the most bioreceptive adhesive, but on the other hand, it is also the most stable adhesive to artificial aging, regarding color alteration, degree of polymerization, and pH (Borges et al., 2018). The use of kudzu starch as an adhesive in the restoration of cultural heritage has shown that kudzu has an optimal chromatic behavior during the accelerated aging process compared to common starch adhesives. On the other hand, daidzein, a natural anti-microbial 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 a cleaner, resistance to biological colonization, and consolidant is promising (Lama et al., 2020). 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-towater ratio, cooking method, cooking duration, cooking temperature, sieving, and storage time or method affect the quality of the end-product for conservation use (Banik et al., 2011; Matsumaru, 2021).

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 adhesive tissue and then tissue paper containing starch paste is activated by water and it is connected to the work by a local press or by using a cold press machine without applying heat. These adhesive tissues are used to restoration works in which ink and colors are not sensitive to water and it does not move due to contact with water. In this method, the durability of the paste will increase and it will be easier to use and also will be economical.

1. Literature Review

Area and Cheradame (2011) depicts a multi-component material, and because of its complex and varied nature, research findings in paper chemistry can be difficult to interpret. Deterioration of paper is caused by many factors, such as acid hydrolysis, oxidative agents, light, air pollution, or the presence of microorganisms. The origin of the cellulosic material, as well as pulping and papermaking procedures, additives, and storage conditions, play a crucial role. The chemical changes occurring within paper thus involve multi-parameter processes.

Alexopoulou and Zervos (2016) determined the degree of

implementation at the international level of the various paper conservation methods found in the literature. Participating organizations in the survey mainly include national libraries, archives, and museums that practice paper conservation. The results of the survey indicate that the types of objects treated by the majority of the participating organizations consist mainly of manuscripts, archival material, books, maps, topographical drawings, and photographic material.

Biricik et al. (2011) determined the changes in strength and printing properties of sulfite base paper and vegetable parchment base paper with sizing by starch having a solid content of 10% on the K-Coater laboratory type. According to the results of paper strengths and printing tests, it has been found that surface sizing with starch improves paper physical strength properties and printability.

Borges et al. (2018) analyzed five adhesives namely starch paste, unsupported ArchibondTM, carboxymethyl cellulose, hydroxypropylcellulose, and methylcellulose for their chemical stability and fungal bioreceptivity (the ability of a material to be colonized by fungi). The bioreceptivity of products used in conservation and restoration is a poorly explored subject, despite its great relevance for the preservation of objects.

Carter (1996) identified the nature of the chromophores found in cellulose, hemicellulose, and lignin. The photooxidation of lignin-containing papers and the mechanism for photoyellowing are discussed. This is followed by a description of the basic principles of conservation bleaching, which involves chemically treating papers in order to remove unwanted discoloration or stains. The washing of paper and the use of oxidizing and reducing bleaches are presented. The discussion on oxidizing bleaches includes hydrogen peroxide, alkaline hypochlorite, chlorine dioxide, and sunlight.

2. Material and Methods

It includes the preparation of adhesive tissue paper using starch paste, the application of the adhesive to paper samples, testing its adhesive properties through tensile strength and adhesion resistance measurements, and the use of FTIR-ATR spectroscopy to analyze the molecular structure of the adhesive tissue paper.

2.1 Materials

The process for obtaining rice starch involves sourcing high-quality rice grains, cleaning and rinsing them to remove impurities, and then soaking the rice in water. Afterward, the rice is milled or ground to extract the starch. The obtained rice starch is then dried and powdered for use as an adhesive material. Additionally, the materials section describes the preparation of samples, which involves applying the rice starch adhesive to paper samples for subsequent testing and evaluation.

2.1.1 Preparation of Rice Starch

In this research, rice starch was prepared according to 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, at a ratio of 3 to 1 (3 parts paste and 1 part water), due to its suitable concentration, was used as a paste for sampling.

2.1.2 Preparation of Samples

Tissue paper (Japanese) 9.10 g/m², 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 paste was gently placed on the tissue paper with a brush, and the papers were covered with the prepared paste. Then they were dried at ambient temperatures. 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.

2.2 Methods

The materials undergo rigorous testing using various

Sample Code	Description of Treatment					
Р	control paper without starch paste					
PS	Paper coated with starch paste					

Table 1. Prepared Samples and their Abbreviated Code

methods, including accelerated aging to simulate longterm effects in a short time, pH determination to assess chemical stability, colorimetry for color analysis, FTIR-ATR spectroscopy to identify molecular structures, tensile strength to measure mechanical properties, and adhesion resistance to evaluate bonding capabilities. These tests ensure the materials' quality and performance under different conditions.

2.2.1 Accelerated Aging

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 90 °C and a relative humidity of 50% for 384 hours. This test was performed using a Memmert oven with a maximum temperature of 120 °C, 600 watts, and 220 volts. In light aging according to ASTM standard D6789-02, the samples were exposed to the 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 30 °C, depending on the distance of the lamp from the samples. After the end of the considered time, the changes made in the samples were compared with those before aging.

2.2.2 pH Determination

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

2.2.3 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. To evaluate the changes in these factors in the samples, Equation 1 is used.

$$\Delta E_{Lob} = \sqrt{\left(L_{2}^{*} - L_{1}^{*}\right)^{2} + \left(a_{2}^{*} - a_{1}^{*}\right)^{2} + \left(b_{2}^{*} - b_{1}^{*}\right)^{2}} \qquad (1)$$

In this equation, L_{1}^{*} , a_{1}^{*} , and b_{1}^{*} demonstrate samples without aging, and L_{2}^{*} , a_{2}^{*} , and b_{2}^{*} demonstrate those after the aging process. Also, also ΔL^{*} , Δa^{*} , Δb^{*} and ΔE^{*} show the total changes in colors in the CIE lab.

2.2.4 FTIR-ATR 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- Fourier Transform Infra-Red (ATR-FTIR) were 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 Attenuated Total Reflection (ATR) of the samples.

2.2.5 Tensile Strength

The tensile strength of the samples was measured before and after aging according to ISO 1924-3. To measure the tensile strength, the paper was cut to 150 mm by 15 mm and placed vertically between the upper and lower jaws of the machine, which was 100 mm 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 and provided in the standard as Equation 2.

$$\sigma_T^b = \frac{\overline{F_T}}{b} \tag{2}$$

 σ_{τ}^{b} is the tensile strength in (kN/m) unit, \overline{F}_{τ} is the average of maximum tensile force in N unit, and b is the width of sample in mm unit.

2.2.6 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.

The test plates were prepared with a width of 25 mm and a length of 305 mm, and 241 mm of their length were connected to each other using a cold press machine. This test was done using the 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 127 mm of the connection line after the first peak of the graph, as shown in Figure 1.

3. Results and Discussion

3.1 pH Changes

In examining the pH 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 the samples after aging remained in the range of 6 and 7. On average, the highest pH level corresponds to the control paper without starch paste after moist-heat aging. The lowest pH is related to the samples covered by starch paste after moist heat aging. The pH of the samples covered by starch paste, after light aging and moist heat aging, has decreased compared to the control samples without paste, as shown in Figure 2. Of course, the decrease in pH is predictable as a result of the accelerated aging process of paper. The decrease in pH due to aging is caused by the release of H+ ions during the hydrolysis of cellulose (Area & Cheradame,



Figure 1. Schematic Design of the Samples Prepared For the Adhesion Resistance Test



Figure 2. The pH of the Samples before and After Aging

2011). Conducting hydrolysis and oxidation reactions leads to the breaking of the cellulose chain and results in the phenomenon of paper acidification. In this condition, the pH of the paper decreases, its color tends to yellow, it becomes very brittle and fragile, and finally the mechanical strength of the paper is lost (Biricik et al., 2011).

3.2 Color Changes

To study the amount of color change in the samples before and after aging, the average change of L* a* b* factors in each sample was calculated. The results are tabulated in Table 2. In Figure 3, the changes in the L* factor (light-dark) of the samples are shown. Based on the obtained results, before aging, the amount of L* factor in PS samples (tissue paper covered by starch paste) decreased compared to P samples (tissue paper without starch paste). Based on this, it can be concluded that the application of starch paste to the paper has caused the color of the paper to darken. In sample P, after moist-heat aging, the L* factor has decreased to a greater extent, and the color of the samples has become slightly darker after aging, but this change has been slight. In the PS sample, the most color changes occurred after light aging. The color changes made in these samples were also very small. Accelerated aging has caused a

Sample Code	Before Aging		Light Aging		Moist-Heat Aging			ΔΕ			
	L*	a*	b*	L*	a*	b*	L*	a*	b*	Light Aging	Moist-Heat Aging
Р	87.58	-1.96	-0.54	87.24	-2.23	0.18	85.88	-2.34	0.36	0.83	1.95
PS	87.1	-2.26	-0.16	86.46	-2.12	-0.52	86.82	-2.3	0.3	0.72	0.52

Table 2. The Average Colorimetric Factors of Samples before and After Aging



Figure 3. Factor L* Changes (light-dark) Of the Samples

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 its darkness increases (Holik, 2006). The yellowing of paper materials and the reduction of their brightness with 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 (Havlinova et al., 2002).

Another factor investigated in color changes is the a* factor, which shows the amount of color change from green to red. In Figure 4, the changes in a* (green-red) of the samples before and after aging are presented. Based on the obtained results, the amount of factor a* in sample P has decreased after aging; this amount of reduction after moist-heat aging is greater than that of light aging, and it shows that the color of the samples has become



Figure 4. Factor a* Changes (green-red) of the Samples

lighter; of course, the amount of this color change was very little. In the PS sample, after moist-heat aging, the amount of factor a* has increased and the color of the sample has become slightly darker.

Figure 5 shows the changes in the b* factor (yellow-blue) of the samples. In sample P, after aging, the b* factor increased so much that this increase after moist-heat aging was greater than that after light aging, and the color of the samples tended to be yellow. In the PS sample, after light aging, the factor b* decreased and the color of the sample became brighter, but after moist heat aging, the amount of this factor increased and the color of the sample became darker. In general, after both stages of aging, the factor b* in the PS sample has decreased compared to the P sample, and the color of the samples tended to be yellow.

The total color change (ΔE) of the samples is shown in Figure 6. The highest value of ΔE belongs to control





Figure 5. Factor b* Changes (Blue-Yellow) of the Samples

Figure 6. The Rate of Color Changes (ΔE) In the Samples

samples without starch paste after moist-heat aging. While the samples covered by starch paste showed the lowest ΔE after moist-heat aging and light aging, respectively. In general, the colorimetric values (L*, a*, and b*) for the samples covered by starch paste were more appropriate than the control samples without starch paste. According to the changes examined in the colorimetric charts, it seems that the color changes created in the samples were caused by the effects of accelerated aging conditions. Because of the aging of the samples, the color changes have also increased. This is caused by the oxidation and acid hydrolysis of cellulose. By breaking glycosidic bonds, acids reduce the degree of cellulose polymerization, which causes the paper to become brittle over time (Missori et al., 2006). The color changes created in control papers without starch paste are the result of chemical destruction or oxidation of anhydroglucose units of cellulose molecules and therefore the presence of secondary groups known as colored chromophores (Carter, 1996; Ďurovlč et al., 1991). In addition, the color changes created after moist heat aging can be the result of the increase of oxygencarrying groups such as carbonyls and carboxylic acids, which are created due to the degradation of cellulose. These groups are chromophores that cause changes in the color of the polymer from colorless to yellow and even brown (Mert, 2008).

3.3 Adhesion Resistance

As seen in Figure 7, the results obtained from the adhesion resistance test show that the adhesion of the samples



Figure 7. Changes in Adhesion Resistance of Samples before and After Aging

coated with starch paste has decreased after light aging and moist heat aging. The adhesion resistance has decreased to a greater extent after moist-heat aging compared to light 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, which leads to a decrease in adhesion resistance. Placing the papers covered by starch paste in conditions of accelerated aging causes 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, the destruction reaction also increases (Anderson & Reidell, 2009).

3.4 Tensile Strength

Figure 8 shows the results obtained from the tensile strength test of the samples. Based on the results, the amount of tensile strength of the samples coated with starch paste has increased compared to the control samples without paste before aging. In other words, starch paste has increased the tensile strength of the samples. After light aging and moist heat aging, the tensile strength of the samples decreased. However, the tensile strength of the samples coated with starch paste has increased compared to the control samples without paste. 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



Figure 8. Changes in the Tensile Strength of the Samples Before and After Aging

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 (Dadmohamadi et al., 2022). In general, the starch paste caused better interaction and mixing of the starch paste and paper fibers, which increased the strength of the samples.

3.5 FTIR-ATR Spectroscopy

In the ATR-FTIR spectrum of the paper sample coated with starch paste, which can be seen in Figure 9, the change in absorption intensity in some areas, such as the increase in absorption in the range of 1670-11680 cm⁻¹, the C=O double bond region, is related to carbonyl groups. After moist-heat aging and light aging, it can be caused by the oxidation of these double bonds, which causes the samples to change color (Hajji et al., 2015). The appearance of a peak in the range of 1100 cm⁻¹, 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 polysaccharide (Silva et al., 2007).

The increase in absorption in the range of 1500–1400 cm⁻¹ due to the asymmetric bending vibration of the CH group indicates the increase of this group, which confirms the transverse destruction (Lukinac et al., 2009). In general, the increase in the intensity of peaks in moist-heat aging was greater than in light aging, and the structural changes created in the samples due to moist-heat aging were greater than in light aging.

Conclusion

In this research, the performance of adhesive tissues made by starch paste was evaluated for the conservation and restoration of paper works. Based on the results obtained after the application of starch paste, the pH value of the samples covered by this paste increased slightly compared to the control samples without paste. After two stages of aging, there was a slight decrease in the pH of the samples compared to the previous stage, but the pH of the samples remained in the range of 6 and



Figure 9. ATR-FTIR Diagrams of Paper Coated By Starch Paste before and After Aging

7. The samples covered by starch paste had the lowest pH after moist-heat aging compared to other samples. The results of the colorimetric test showed that the samples coated with starch paste had fewer color changes than the control samples without paste. The samples covered by starch paste showed the lowest amount of color change after moist heat aging and light aging, respectively. The data obtained from the tensile strength test of the samples showed that the tensile strength of the samples coated with starch paste increased compared to the control samples without paste. After light aging and moist heat aging, the tensile strength of the samples decreased compared to the previous stage, but the tensile strength of the samples coated with starch paste increased compared to the control samples without paste. The results of the adhesion resistance test of the samples showed that the adhesion of the samples decreased after light aging and moist heat aging. The amount of adhesion in the samples has decreased to a greater extent after moist-heat aging compared to light aging.

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