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The Evaluation of the Effect of Agarose-Based Cleaning Systems on Cotton Fabrics

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Abstract

Cotton textiles, commonly affected by ink stains, due to frequent handling and misuse, present a significant conservation challenge. As interest in sustainable conservation methods increases, agarose-based gels have gained attention as promising, non-invasive alternatives due to their controlled application and minimal impact on the treated material. This study investigates the efficiency of agarose gel-based systems in comparison to conventional solvent-based treatments for removing ink stains from both aged and unaged 100% cotton fabrics. Various concentrations of agarose gels (2%, 3%, and 5%) combined with different ratios of isopropyl alcohol (60%, 80%, 100%) were evaluated alongside commercial and solvent-based treatments—both with and without suction assistance. The efficacy of these treatments was assessed through colorimetric measurements, visual inspection, USB microscopic inspection, FTIR spectroscopy, and mechanical testing. The results indicated that although solvent-based treatments with suction—especially those using 100% isopropyl alcohol—were effective in removing stains, they notably weakened the fabric. In contrast, agarose-based systems, particularly the 3% concentration combined with 60% and 80% isopropyl alcohol, achieved a balanced outcome by providing moderate cleaning while maintaining the fabric's structural strength and chemical stability. These findings underscore the potential of gel-based systems as safer and effective options for conserving historical textiles.

Keywords: Ink stains; gel-based systems; solvents; visual and microscopic inspection; colorimetric measurements; FTIR; mechanical properties

1-Introduction

Historical textiles provide valuable insights into social history, international trade, agricultural development, artistic trends, and technological advancements. Accordingly, preserving and passing down these fabrics to future generations is crucial [1]. Museum textiles are considered sensitive materials for deterioration by various deterioration factors such as the surrounding environment, improper handling and varied use [2], [3]. As a result, they are susceptible to a variety of stains [3]. This research paper addresses the removal of ink stains, a very common and persistent issue in textile collections [4].

Ink is defined as a liquid or viscous material used in writing and printing. Its formulations have become so complex that nearly a million new formulas emerge each year. In this context, studying a particular type of ink necessitates characterizing its chemical composition as precisely as possible [5]. Ink is typically categorized into three prominent types: iron gallotannate ink (also known as iron gall ink), Chinese ink, and printing ink. Ink was first used by the Chinese, who mixed black stone powder with lake pigments. However, true ink as we know it emerged in the 3rd century BCE [6]. Modern ink formulations contain many chemicals aimed at improving ink quality. Modern inks are complex mixtures that include numerous chemicals and colorants, which provide the desired hue [7], [8]. Water or organic solvents are used as a medium for dissolving or dispersing pigment or dye to bind the ink to the substrate. Humectants are used to prevent premature drying, while surfactants control the film thickness formed by the ink on the surface [9], [10]. Inks are classified as dye-based or pigment-based. Similarly, the solvents used in their manufacturing classify inks into water-based and solvent-based inks. Additional chemical reagents used in pigment-based inks ensure strong adhesion of coarse pigment particles to the substrate [11] and prevent detachment through mechanical abrasion. These components include typical resins in solvent-based inks or binding agents in water-based inks. Ballpoint pens were first introduced in 1939 abroad and in 1945 in the United States. The ink in ballpoint pens underwent several changes to refine the pens' functionality. In 1950, ballpoint ink shifted from oil-based to glycol-based to reduce smudging. Metallic pigments like copper phthalocyanine blue-green are widely used due to their solubility. Today, copper phthalocyanine is preferred as a cyan pigment worldwide, acting as a soluble dye or an inorganic pigment. It contains heavy metallic copper, making it easily detectable in ink samples [12]. Conversions occur in inks on textile surfaces when solvents begin to evaporate, dyes degrade, and resins polymerize [13], [14]. Certain precautions remain necessary for conservators. For example, dyes degrade when exposed to light and high temperatures, making storage conditions crucial to preserving ink longevity [15].

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stains are very challenging for conservators. A dissolved colorant or dye used in ink manufacture will penetrate the surface of porous materials as a stain [16]. Stains are a result of a chemical reaction between the staining substance and the fabric. Stains primarily form when a substance is spilled onto a surface and the molecules are trapped inside the fibers and pores of the material. The spilled substance coats the underlying material, and the newly formed stain reflects back the light of its own color. Water swells natural fibers (e.g., cotton), accordingly a water-based ink stain will go deeper into a natural fiber unless a special hydrophobic finish (i.e., water repellent) has been applied [17]. [18] The presence of stains not only affects the appearance and aesthetics of objects but also impacts their functionality and durability [19]. These stains present a form of damage that is resistant to traditional conservation treatments such as solvent cleaning due to the absorbent nature of natural fabrics (e.g., cotton) and the way in which dye-based materials interact with the surface of the fabric [16]. To address this issue, stain removers have become essential tools in cleaning procedures, offering effective solutions for removing stains [18]. However, while stain removers serve their intended purpose, their use raises concerns about their effects on the objects. Many traditional stain removers contain chemicals that can harm the artifacts and the environment, leading to environmental pollution and health risks [20], and they are often flammable [21], and poorly controllable [22].

Increased awareness of the harmful effects of chemicals on the environment and human health has led to the discovery of more sustainable and eco-friendly alternatives [23]. Gelled systems are considered the most environmentally friendly and sustainable cleaning solutions [24]. A gel is a polymer network that can absorb a large quantity of solvent and swell due to a physical or chemical stimulus [25]. To create a gel, two essential components are required: the gelling agent and solvent. Other components, such as surfactants and pH buffers, are used to either make the solvent and gelling agent compatible or to modify the gel's effect on the solute [26]. Using a proper gelled system is a simple, practical, and non-invasive method for conservators to reduce damage to objects by releasing liquid cleaning agents in a controlled manner. It ensures simple application and removal [27]. Gel systems with solvents of different polarities are necessary for removing foreign compounds such as ink stains. Organic solvents, water, enzymes, and chelating solutions can be gelled [28]. To circumvent the drawbacks of conventional fluid gels, so-called rigid gels such as gellan gum and agarose-based gels have been utilized. These gels do not require after-treatment due to their physical form and their limited adhesive strength [29].

In view of that, this study aimed at evaluating different cleaning procedures (i.e., solvent-based and gel-based systems) to remove ink stains (i.e., unaged set-in pen ink stains and aged pen ink stains) from cotton textiles. The efficacy of selected treatments was evaluated in terms of the efficiency of stain removal, absence of aesthetic alteration, and long-term effects on the physical, optical, and chemical properties of cotton textiles. A multi-analytical approach was conducted during this study using visual inspection, digital microscopy, Fourier transform infrared spectroscopy (FTIR), colorimetric measurements, and mechanical testing.

1. Experimental

1.1. Materials

Table 1: Selected cleaning treatments

No.	Treatment	Treatment description
1		Agarose 2% with Isopropyl alcohol 60%
2	Agarose 2% with Isopropyl alcohol	Agarose 2% with Isopropyl alcohol 80%.
3		Agarose 2% with Isopropyl alcohol 100%.
4		Agarose 3% with Isopropyl alcohol 60%
5	Agarose 3% with Isopropyl alcohol	Agarose 3% with Isopropyl alcohol 80%.
6		Agarose 3% with Isopropyl alcohol 100%.
7		Agarose 5% with Isopropyl alcohol 60%
8	Agarose 5% with Isopropyl alcohol	Agarose 5% with Isopropyl alcohol 80%.
9		Agarose 5% with Isopropyl alcohol 100%.
10		Isopropyl 60%,
11	Isopropyl alcohol	Isopropyl 80%,
12		Isopropyl 100%,
13		Isopropyl 60%,
14	Isopropyl alcohol under suction table	Isopropyl 80%,
15		Isopropyl 100%,
16	Commercial solvent	Without use of suction table
17		Under suction table
18		Agarose 2% with commercial solvent
19	Agarose with commercial solvent	Agarose 3% with commercial solvent
20		Agarose 4% with commercial solvent

The samples used in this study are unprocessed, beige 100% cotton samples. The twill pattern of the fabric is 1/3. The weight per square meter is 200 g, the air permeability is 16.40 cm^3/cm^2, the pressure is 125 pa, the area is 5 cm2, the tensile is 590 N, the elongation is 21.69%. The samples were washed at a temperature of 60 °C for 30 minutes to remove the starching materials suspended in the material.

Two sets of samples were prepared. The first set is aged cotton samples stained with unaged set-in pen ink stains; while the second set is aged cotton samples stained with aged fountain pen ink stains. Artificial aging was performed to assess the long-term effects of the treatments listed in (Table 1). The solvent, Inko Go, is an Egyptian product designed for removing ink stains from white and colored fabrics and is certified under ISO 9001:2008 and ISO 14001:2004. Inko Go is a mixture of organic solvents and non-ionic materials.

1.2. Methods

1.2.1. Accelerated aging

Samples were artificially aged at a temperature of 105 °C and relative humidity of 65% RH for 22 days, equivalent to 100 years [30].

1.2.2. Preparation and application of agarose gel pads

The gel cleaning method using aqueous cleaning solutions involves two key principles: firstly, incorporating a gelling agent into the cleaning solution to enhance its viscosity and improve control during application on a surface; and secondly, enhancing the cleaning efficiency of water by introducing chelating agents, pH buffers, and/or enzymes or surfactants [30]. The polysaccharide agarose is produced by the red seaweed Gelidium and is commonly used in agarose gel electrophoresis [32]. Agarose gel is created by scattering powdered agarose in an aqueous solution and then heating it to a temperature higher than 85 °C. Gelation occurs when the temperature drops below 40 °C [33]. The agarose gel's porous nature enables the gradual movement of liquids by capillary action. The absorption rate of the gel can be altered by adjusting the concentration of agarose in the solution. At elevated concentrations, the pores exhibit reduced size, and the capillary action is enhanced; conversely, at lower concentrations, the reverse effect occurs [32].

The agarose gel was prepared in three concentrations (i.e., 2, 3, 5%). A weighted amount of agarose powder was placed into 100 ml of water, and then the vessel was heated to 85 ºC using an electric heater. After heating, each concentration was poured into three non-stick molds. Next, a measured amount of solvent was added to the agarose sol-gel and stirred it to achieve homogeneity. The agarose-based formulations were allowed to cool into a rigid gel form with a thickness of 0.5 cm. The resultant sheets of rigid agarose gels were cut into squares with a size of 4 ×1 cm^2 to allow for a comparable surface coverage. The gel pads were placed on the stain with a weight on top (i.e., 5 g) [34]. Three different treatment durations were used (i.e., 30, 60, or 120 minutes). This procedure was performed at the National Institute of Standards (NIS) in Cairo.

1.2.3. Evaluation methods

1.2.4. Chromatic change measurements

The color values in the CIE lab system were measured with an Optimatch 3160 from the SDL Company, the National Institute of Standards (NIS) in Cairo, Egypt. The untreated and treated samples were measured before and after artificial ageing and compared to the control sample (i.e., the blank sample). The total color difference ΔE and the CIELAB color parameters (L*, a*, b*) were used, where L* defines lightness and varies from 0 (black) to 100 (white); a* represents the red/green axis, where +a means red and –a means green; b* represents the yellow/blue axis, where +b means yellow and –b means blue. All values of L*, a*, and b* were obtained before treatment and after treatment [35], [36].

1.2.4.1. Visual inspection

This preliminary procedure is very effective in documenting the resultant changes post treatment.

2.2.3.3. USB digital microscope

A professional portable handheld mobile LCD digital microscope with MP digital zoom 100–1,000 was used to study the treated textile morphology. This procedure was performed at the Laboratory at the National Institute for Standards (NIS), Cairo, Egypt.

2.2.3.4. Fourier transforms infrared spectroscopy analysis

FT-IR spectroscopy was used to study the chemical changes that may have occurred after artificial aging and after treatment. The spectra were obtained in absorbance mode using a Nicolet 380 Ft-IR Spectrometer, in the frequency range of 4000–400 cm^{-1}. The analysis was carried out at the National Institute of Standards (NIS) in Cairo, Egypt.

2.2.3.5. Mechanical testing: tensile breaking strength analysis

The tensile strength and elongation of untreated and treated samples were measured by using the dynamometer produced by SDL ATLAS, H5KT. The untreated and treated samples were kept in optimal conditions for 24 h; the test was done in a standard atmosphere, i.e., at a temperature of 19 °C and 64% RH. The range of load was 100 N, the extension range was 10 mm, the gauge length was 100 mm, the preload was 1.0 N and the speed was 100 mm/min. Measurements were carried out at the metrology laboratory of the National Institute of Standards (NIS) in Cairo, Egypt.

2. Results and discussion

2.1. Chromatic change measurements

Table 2: L, a, b, and ΔE values for the samples soiled with unaged set-in stains

Samples	L	a	B	ΔE
Before cleaning	69.05	2.83	3.40	-
Agarose 2% with Isopropyl 60%	72.10	0.78	5.08	10.59
Agarose 2% with Isopropyl 80%	72.25	2.32	6.71	11.19
Agarose 2 % with Isopropyl 100%.	73.36	0.25	3.70	7.59
Agarose 3% with Isopropyl 60%	68.94	0.36	9.55	12.34
Agarose 3% with Isopropyl 80%	69.27	7.62	7.11	11.89
Agarose 3% with Isopropyl 100 %	74.99	2.64	1.41	9.16
Agarose 5 % with Isopropyl 60%	70.52	0.53	5.87	11.99
Agarose 5% with Isopropyl 80%	72.11	2.27	2.29	9.22
Agarose 5% with Isopropyl 100%	71.87	2.42	1.25	9.34
Isopropyl 60%	73.20	2.07	2.43	7.46
Isopropyl 80%	77.13	0.70	6.78	7.64
Isopropyl 100%	71.19	1.26	4.54	6.27
Isopropyl 60% under suction table	72.25	1.78	1.99	6.80
Isopropyl 80% under suction table	71.88	1.90	2.74	7.15
Isopropyl 100% under suction table	78.38	1.34	12.04	15.07
Commercial Solvent	73.35	2.80	1.05	8.41
Commercial Solvent under suction table	77.66	1.73	12.24	13.59
Commercial Solvent with Agarose 2%	75.87	1.43	1.68	9.66
Commercial Solvent with Agarose 3%	71.97	1.31	4.56	11.41
Commercial Solvent with Agarose 5%	73.57	1.30	5.36	9.29

Table 3: L, a, b, and ΔE values for the samples soiled with aged stains

Samples	L	a	B	ΔE
Before cleaning	76.77	0.68	5.63	-
Agarose 2% with Isopropyl 60%	76.98	0.32	5.03	7.90
Agarose 2% with Isopropyl 80%.	77.42	0.40	4.55	7.49
Agarose 2 % with Isopropyl 100%.	75.39	0.24	6.32	6.19
Agarose 3% with Isopropyl 60%	76.68	0.71	5.79	7.44
Agarose 3% with Isopropyl 80%	73.00	0.79	2.04	9.85
Agarose 3% with Isopropyl 100 %	76.04	0.14	5.89	10.05
Agarose 5 % with Isopropyl 60%	73.74	0.10	3.16	6.90
Agarose 5% with Isopropyl 80%	74.98	0.25	5.79	5.21
Agarose 5% with Isopropyl 100%	75.47	0.44	5.54	6.55
Isopropyl 60%	74.07	0.16	6.57	10.28
Isopropyl 80%	75.07	0.45	5.46	11.35
Isopropyl 100%	71.57	0.28	4.51	6.17
Isopropyl 60% under suction table	73.67	0.67	5.91	8.69
Isopropyl 80% under suction table	76.67	1.02	10.18	11.34
Isopropyl 100% under suction table	74.01	0.68	7.18	8.78
Commercial Solvent	75.18	0.37	7.30	7.67
Commercial Solvent under suction table	76.29	1.85	12.06	12.60
Commercial Solvent with Agarose 2%	77.34	0.16	4.93	10.51
Commercial Solvent with Agarose 3%	78.62	0.08	3.45	9.93
Commercial Solvent with Agarose 5%	79.55	0.27	7.59	8.12

Chromatic change measurements for the unaged set-in pen ink stains showed that best results were obtained in the case of isopropyl alcohol 100% under the suction table, followed by the commercial solvent under the suction table, followed by agarose 3% with isopropyl 60%, followed by agarose 5% with isopropyl 60%, followed by agarose 3% with isopropyl 80%. And the least efficient treatment was isopropyl alcohol 100%. This is detected from the large color change difference (ΔE) that resulted from the difference in color between the original stain color and the cotton surface revealed post cleaning. As for the chromatic change measurements for the aged pen ink stains, the best results were obtained in the case of the commercial solvent under suction table, isopropyl alcohol 80%, isopropyl alcohol 80% under suction table and commercial solvent with agarose 2%, and agarose 3% with isopropyl alcohol 100%. And the least efficient treatment was agarose 5% with isopropyl alcohol 80%. Only these treatments will be further tested. Chromatic change results are listed in Table 2 and Table 3.

2.2. Visual inspection

2.2.1. Unaged set-in pen ink stains

Samples treated with agarose 3% and isopropyl alcohol 80%, agarose 3% and isopropyl alcohol 60%, and agarose 5% and isopropyl alcohol 60% showed no visible change post treatment for a period of 30 minutes with regard to stain removal (Fig. 1b), (Fig. 2b), and (Fig. 3b) compared to the control samples (Fig. 1a), (Fig. 2a), and (Fig. 3a), respectively. This implies that the ink was not sufficiently mobilized from the surface by the limited contact time and comparatively mild s olvent strength, particularly when applied through a gel medium that naturally inhibits solvent release [31], [32]. For all samples, the cleaning efficiency slightly increased after treatment for a period of 60 minutes (Fig. 1c), (Fig. 2c), and (Fig. 3c). Better results were obtained after treatment for a period of 120 minutes (Fig. 1d), (Fig. 2d), and (Fig. 3d). This can be attributed to the prolonged interaction between the solvent and the stain facilitated by the gel [33]. This time-dependent effect indicates that gel systems may be effective for gentle cleaning but require adequate exposure to achieve noticeable results. Notably, samples treated with isopropyl 100% under suction table and commercial solvent under suction table exhibited excellent cleaning results, with the commercial solvent providing slightly superior results (Fig. 4b) and (Fig. 5b), compared to the control samples (Fig. 4a) and (Fig. 5a), respectively, likely due to its optimized formulation. On the other hand, samples treated with isopropyl 100% showed the smearing of the stain post treatment (Fig. 6b) compared to the sample before treatment (Fig. 6a), highlighting the risk of unassisted solvent application and underscoring the importance of controlled extraction methods in conservation treatments [33].

Fig. 1. The samples treated with agarose 3% with isopropyl alcohol 80% treament. a) before treatment; b) 30 minutes; c) 60 minutes; d) 120 minutes.

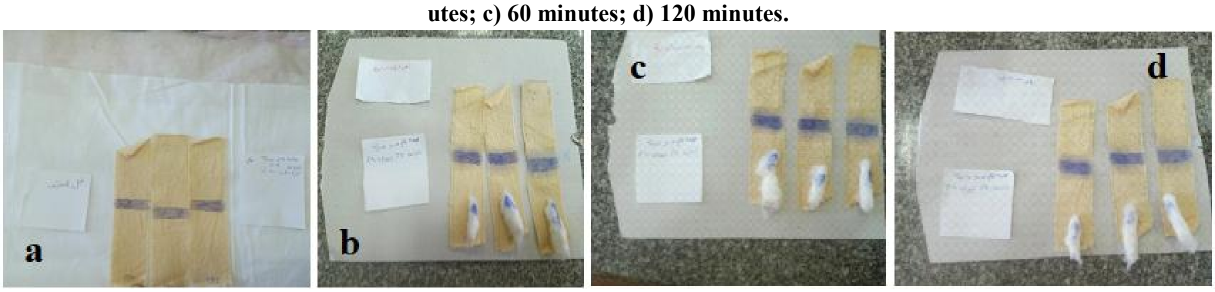

Fig. 2. The samples treated with agarose 3% with isopropyl alcohol 60% treament. a) before treatment; b) 30 minutes; c) 60 minutes; d) 120 minutes.

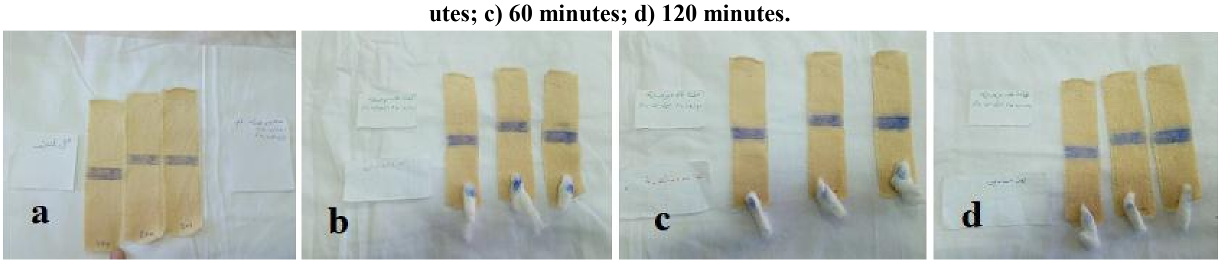

Fig. 3. The samples treated with agarose 5% with isopropyl alcohol 60% treament. a) before treatment; b) 30 minutes; c) 60 minutes; d) 120 minutes.

Fig. 4. The samples treated with isopropyl 100% under suction table. a) before, and b) after treatement.

Fig. 5. The samples treated with commercial solvent under suction table. a) before, and b) after treatement.

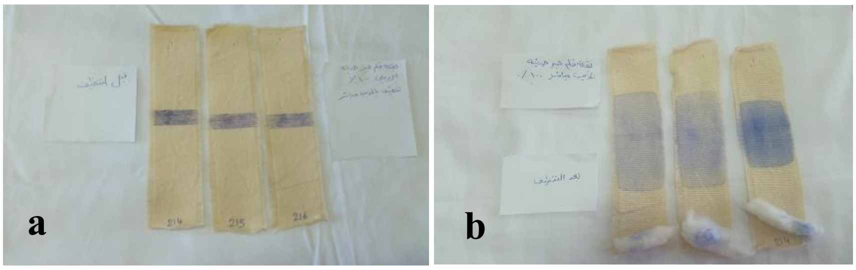

Fig. 6. The samples treated with isopropyl 100% . a) before, and b) after treatement.

2.2.2. Aged pen ink stains

Fig. 7. The samples treated with agarose 3% with isopropyl alcohol 100% treament. a) before treatment; b) 30 minutes; c) 60 minutes; d) 120 minutes.

Samples treated with agarose 3% and isopropyl alcohol 100%, commercial solvent and agarose 2%., agarose 5%, and isopropyl 80% for 30 minutes gave a very poor cleaning effect showing no change (Fig. 7b), (Fig. 11b), and (Fig. 12b) compared to the control samples (Fig. 7a), (Fig. 11a) and (Fig. 12a), respectively. This reflects the increased resistance of aged inks, which likely penetrate deeper into the cotton fibers and undergo chemical changes such as oxidation, making them less responsive to solvent-based cleaning [34], [35], [36]. Slight change with regard to stain removal was observed in samples treated for 60 minutes (Fig. 7c), (Fig. 11c), and (Fig. 12c). Treatment for 120 minutes increased the cleaning efficiency (Fig.

7d), (Fig. 11d), and (Fig. 12d), indicating that extended treatment times are necessary to overcome the tenacity of aged ink residues. Samples treated with isopropyl 80% gave good results (Fig. 8b) compared to the sample before treatment (Fig. 8a). Better results were obtained in the case of the samples treated with isopropyl 80% under suction table (Fig. 9b), which improved solvent action and stain extraction. The most successful results were achieved with the commercial solvent applied under a suction table (Fig. 10b), which effectively removed the aged ink without damaging the sample, in contrast to the samples before treatment (Fig. 9a) and (Fig. 10a). These findings highlight the value of combining moderate solvent strength with mechanical support to address aged stains and emphasize that commercial formulations designed for conservation offer enhanced performance, particularly when paired with controlled application techniques such as suction tables [37], [38].

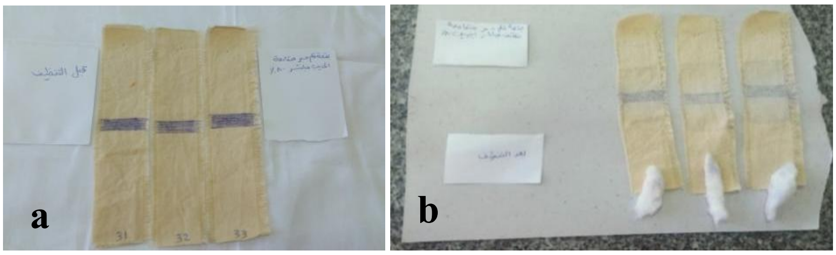

Fig. 8. The samples treated with isopropyl 80% . a) before, and b) after treatement.

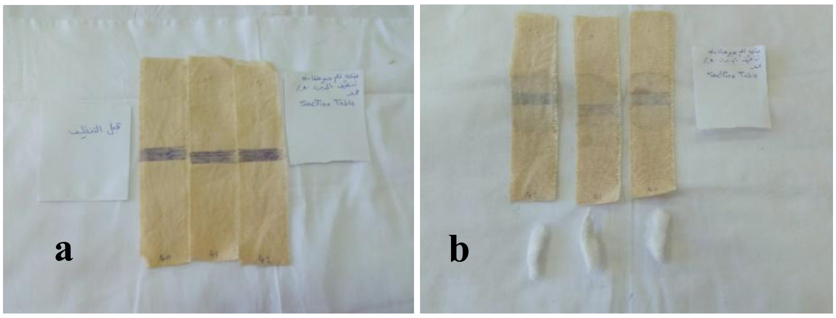

Fig. 9. The samples treated with isopropyl 80% under suction table . a) before, and b) after treatement.

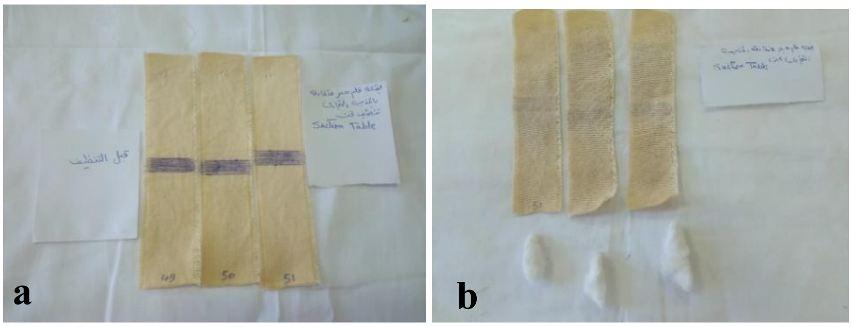

Fig. 10. The samples treated with commercial solvent under suction table . a) before, and b) after treatement.

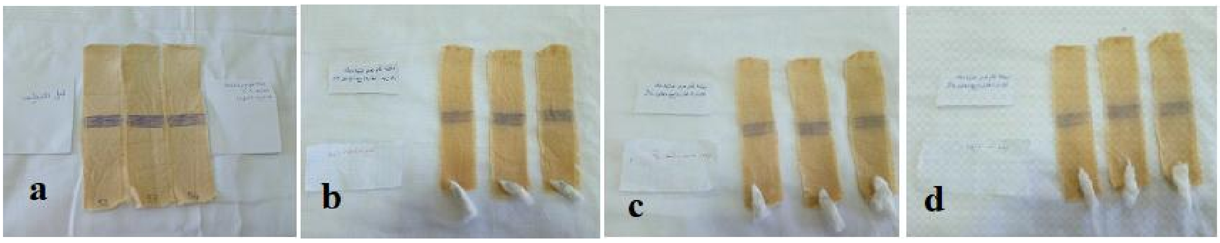

Fig. 11. The samples treated with commercial solvent with agarose 2%. a) before treatment; b) 30 minutes; c) 60 minutes; d) 120 minutes.

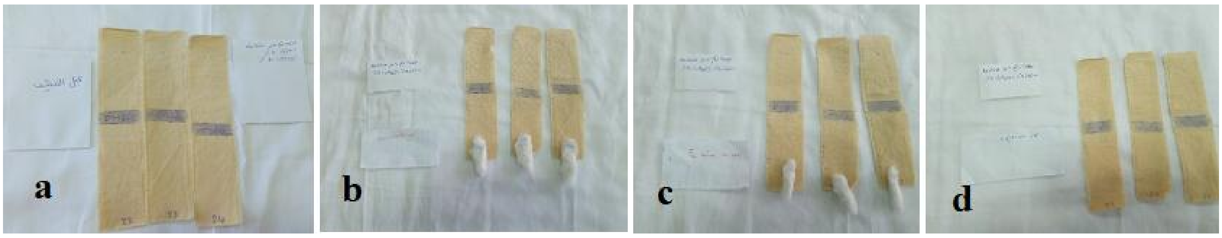

Fig. 12. The samples treated with agarose 5% and isopropyl 80%. a) before treatment; b) 30 minutes; c) 60 minutes; d) 120 minutes.

2.3. USB digital microscope

2.3.1. Unaged set-in pen ink stains

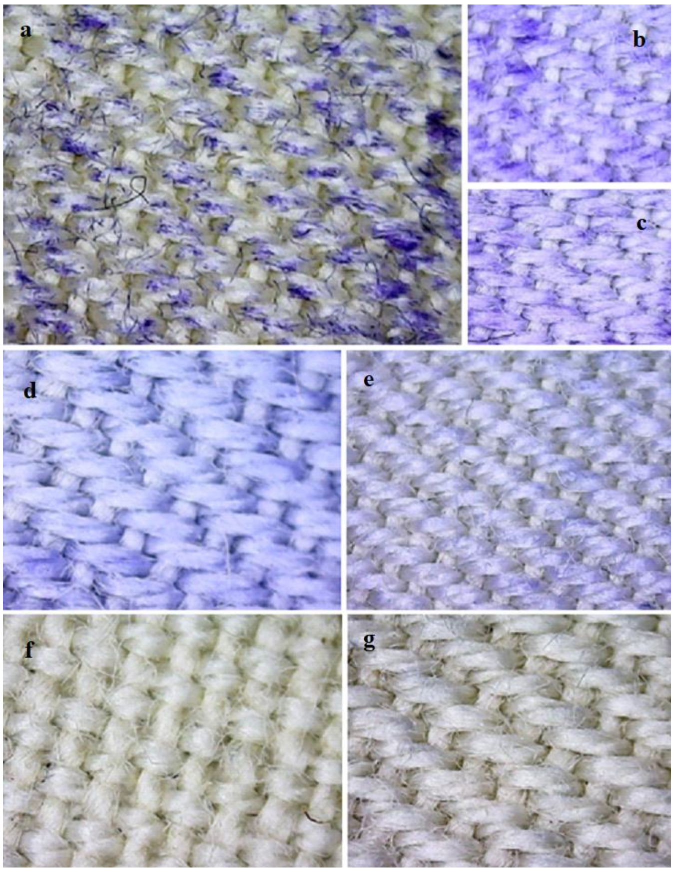

Fig. 13. Microscopic examination of stained control sample (a), the sample treated with agarose 5% and isopropyl alcohol 60% (b), the sample treated with agarose 3% and isopropyl alcohol 60% (c),the sample treated with agarose 3% and isopropyl alcohol 80% (d), the sample treated with isopropyl alcohol 100% (e), the sample treated with the commercial solvent under suction table (f), and the sample treated with the isopropyl alcohol 100% under suction table (g).

Fig. 13a serves as the control sample. The sample treated with agarose 5% and isopropyl alcohol 60% showed considerable ink smearing (Fig. 13b), indicating that the high gel concentration likely restricted solvent diffusion and prevented effective stain removal, while simultaneously mobilizing the ink without adequate extraction [39]. This uncontrolled spreading suggests that a more balanced interaction between the gel and solvent is needed to avoid secondary staining. In contrast, the sample treated with agarose 3% and isopropyl alcohol 60% (Fig. 13c) exhibited less smearing, implying that decreasing the gel concentration facilitated better solvent release and limited the ink's lateral movement. A more favourable outcome was observed in the sample treated with agarose 3% and isopropyl alcohol 80%, which showed satisfactory cleaning efficiency with no ink smearing (Fig. 13d), reflecting that the increased alcohol concentration enhanced ink solubilization while the lower gel content allowed better control over the solvent's action. Further improvements were observed with the direct application of 100% isopropyl alcohol (Fig. 13e), which achieved effective cleaning. However, the absence of a mechanical removal system could still pose a risk of uneven solvent action or partial re-deposition. The sample treated with the commercial solvent under the suction table (Fig. 13f) demonstrated very good results, highlighting the effectiveness of a customized solvent mixture in conjunction with active extraction. The most effective outcome was achieved with the sample treated using 100% isopropyl alcohol under the suction table (Fig. 13g), resulting in outstanding cleaning performance. This highlights the importance of mechanical support, such as suction, in improving solvent effectiveness and reducing smearing, highlighting the need to optimize both chemical and physical cleaning methods for safe and efficient stain removal.

2.3.2. Aged pen ink stains

Fig. 14a represents the control sample. The samples treated with isopropyl alcohol 80% under the suction table (Fig. 14b) and isopropyl alcohol 80% (Fig. 14c) showed good cleaning results, suggesting that this concentration is effective in solubilizing the ink stain without causing significant damage or smearing. The samples treated with agarose 3% and isopropyl alcohol 100% (Fig. 14d) and agarose 5% with isopropyl alcohol 80% (Fig. 14e) showed very good cleaning efficiency. The best results were observed in the samples treated with commercial solvent and agarose 2% (Fig. 14f) and commercial solvent under the suction table (Fig. 14g). These findings demonstrate the superior performance of commercial formulations specifically designed for conservation purposes, likely due to optimized solvent composition and the presence of additives that enhance stain solubilization and surface activity. The use of agarose gel or suction table further improved control and safety during application. Collectively, these results highlight the importance of selecting the appropriate solvent concentration, delivery method (gel or direct), and mechanical support (such as suction) to achieve effective and safe cleaning of ink-stained cotton-based artifacts.

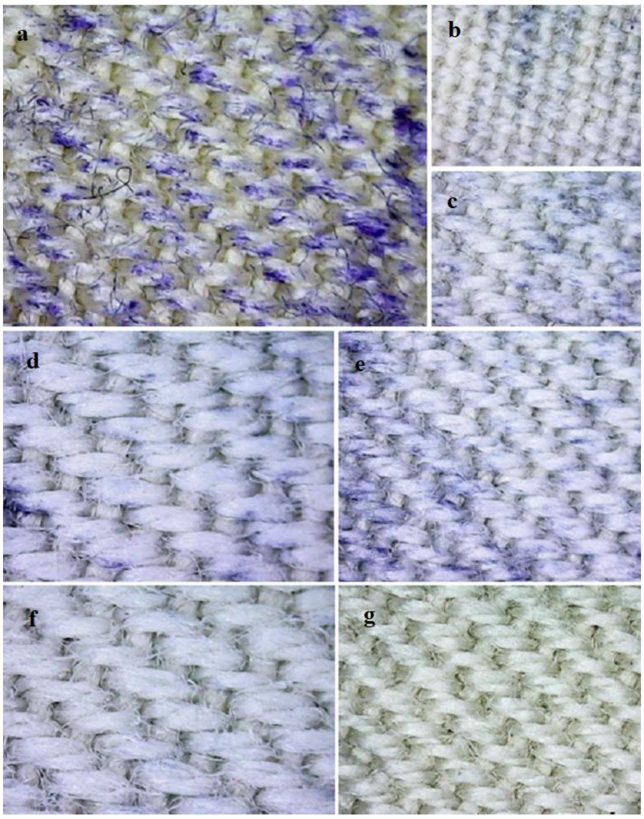

Fig. 14. Microscopic examination of stained control sample (a), the samples treated with isopropyl alcohol 80% under suction table (b), the sample treated with isopropyl alcohol 80% (c), the samples treated with agarose 3% and isopropyl alcohol 100% (d), the sample treated with agarose 5% and isopropyl alcohol 80% (e), the samples treated with commercial solvent and agarose 2% (f), and the sample treated with commercial solvent under suction table (g).

2.4. Fourier transform infrared spectroscopy

Cellulose gives rise to OH stretching vibrations located at 3300 – 3400 cm^{-1}, CH stretching vibrations at 2850 to 3000 cm^{-1}, a weak band assigned to H_2O at ~1645 cm^{-1}, OH bending bands at 1200-1400 cm^{-1}, C-H bending bands at 1350 – 1480 cm^{-1}, C-O vibrations att1000 – 1300 cm^{-1}, CH rocking vibrations at 700 – 900 cm^{-1} [40]. Oxidation of cellulose results in the creation of C=O vibrations at ~ 1645 cm^{-1}. Carbonyl groups are chromophores; these are responsible for the yellowing of textile as its ages [41]. Furthermore, oxidation induces depolymerization of the cellulose, as a result the mechanical strength of the material decreases [42]. As cellulose oxidizes, its hydration capacity decreases as there will be fewer hydroxyl groups to form hydrogen bonds. Consequently, a smaller peak indicates that a portion of the cellulose has degraded [43]. The band at around 1420 - 1430 cm−1 (i.e., the crystallinity band) is associated with the amount of the crystalline structure of the cellulose, while the band at 897 cm−1 is assigned to the amorphous region in cellulose [44], [45]. The increase in the intensity and broadness of the OH vibrations indicate the occurrence of cellulose hydrolysis [40].

2.4.1. Unaged set-in pen ink stains

Table 4: Functional groups for the samples with the unaged set-in pen ink stain post treatment compared to the control sample

Samples	Functional groups (cm^{-1})					
	OH stretching band	CH stretching band	H-O-H band + C=O stretching band	CH bending band + OH bending band	C-O band	CH rocking band
Control sample	3423.48	2901.10	1641.36	1432.22-1334.79	1163.61-1031.34	616.78
Agarose 3% and isopropanol 80%	3404.88	2901.82	1644.19	1429.44-1355.92	1166.57-1030.91	612.64
Agarose 3% and isopropanol 60%	3402.05	2901.82	1647.01	1432.22-1350.27	1163.47-1030.91	612.64
Agarose 5% and isopropanol 60%	3407.07	2901.82	1647.01	1435.05-1350.27	1163.74-1030.91	612.64
Isopropanol 100%	3416.18	2901.82	1641.36	1429.40-1353.09	1160.91-1030.91	615.46
Isopropanol 100% under suction table	3413.36	2901.82	1647.01	1435.05-1347.44	1166.57-1030.91	612.64
Commercial solvent under suction table	3410.35	2901.28	1644.19	1432.22-1350.27	1163.74-1028.09	612.64

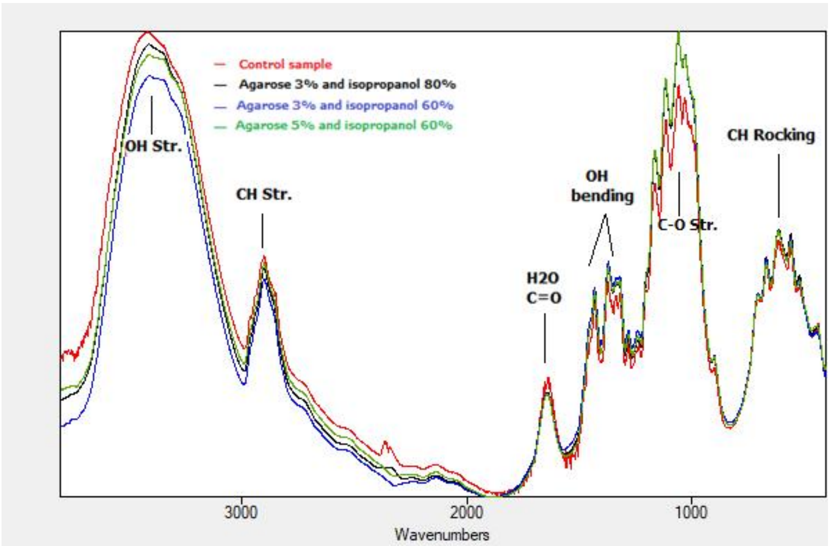

Fig. 15. FTIR spectra for agarose-based cleaning systems compared to the control sample.

Based on previous studies, samples showed an increase in the intensity and broadness of the OH band indicating the occurrence of slight hydrolysis. This change suggests increased hydrogen bonding or disruption of the cellulose structure, likely due to moisture uptake or solvent interactions with hydroxyl groups [46]. Additionally, a slight increase in the intensity of the carbonyl (C=O) band was observed, indicating the occurrence of oxidation [47]. Notably, the degree of oxidation was greater in the case of the sample treated with isopropyl alcohol 100% and the commercial solvent under suction table, implying that these more aggressive treatments, while effective for stain removal, may pose a higher risk to the chemical stability of cotton fibers. Furthermore, the sample treated with isopropyl alcohol 100% under suction table showed a decrease in the crystallinity of cellulose reflecting a loss of structural order, suggesting that the treatment caused some degree of degradation of the cellulose chains [48], potentially due to mechanical stress combined with solvent action. In contrast, samples treated using agarose gel systems exhibited only minor chemical changes, with negligible shifts in the OH and C=O bands and minimal effect on crystallinity. Results are shown in Table 4 and (Fig. 15), (Fig. 16), (Fig. 17), (Fig. 18).

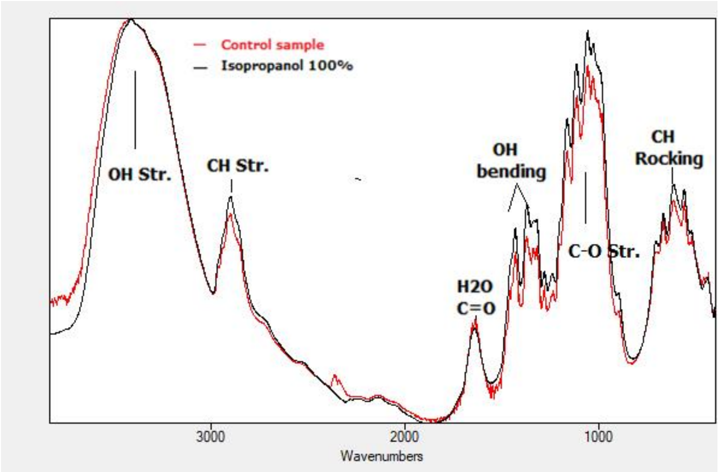

Fig. 16. FTIR spectrum for the sample treated with isopropyl alcohol 100% compared to the control sample.

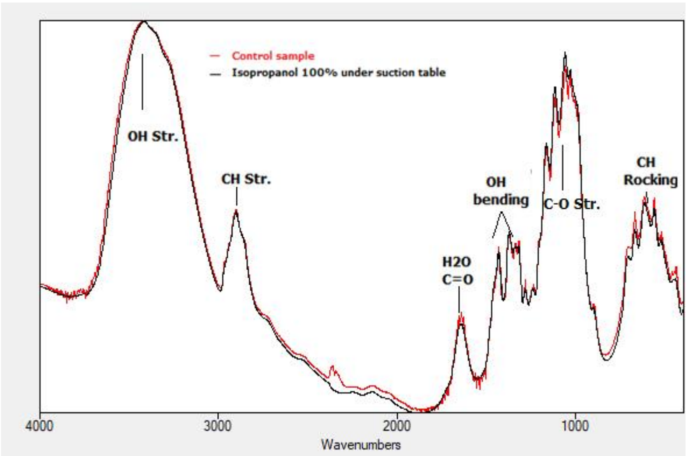

Fig. 17. FTIR spectrum for the sample treated with isopropyl alcohol 100% under suction table compared to the control sample.

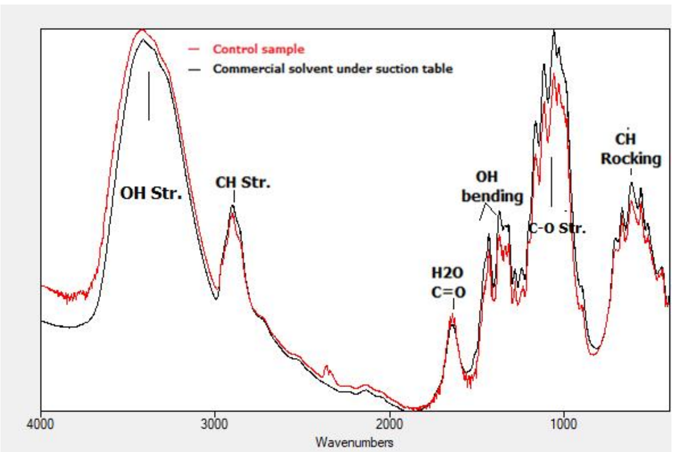

Fig. 18. FTIR spectrum for the sample treated with commercial solvent under suction table compared to the control sample.

2.4.2. Aged pen ink stains

Treatments with 5% agarose and 80% isopropanol, as well as 80% isopropanol led to a noticeable decrease in cellulose crystallinity, indicating partial disruption of the ordered cellulose structure likely due to solvent interaction with the hydrogen-bonding network. Samples treated with 3% agarose and 100% isopropanol showed evidence of slight hydrolysis, as reflected by an increased intensity in the OH absorption band, suggesting limited cleavage of glycosidic bonds or enhanced water affinity within the cellulose matrix. The use of 80% isopropanol under a suction table induced both oxidation and hydrolysis, demonstrated by increase in the vibrations corresponding to the carbonyl (C=O) and hydroxyl (OH) groups, respectively. This implies that while suction-assisted cleaning improves stain removal, it may also accelerate chemical changes in cotton textiles. A very mild oxidative effect was detected in the sample treated with commercial solvent combined with 2% agarose, suggesting a relatively safe chemical profile for this cleaning system. The application of the commercial solvent alone resulted in insignificant chemical changes, indicating its stability and effectiveness as a safe cleaning agent for conservation purposes. Results are shown in (Fig. 19), (Fig. 20), (Fig. 21), (Fig. 22), (Fig. 23) and Table 5.

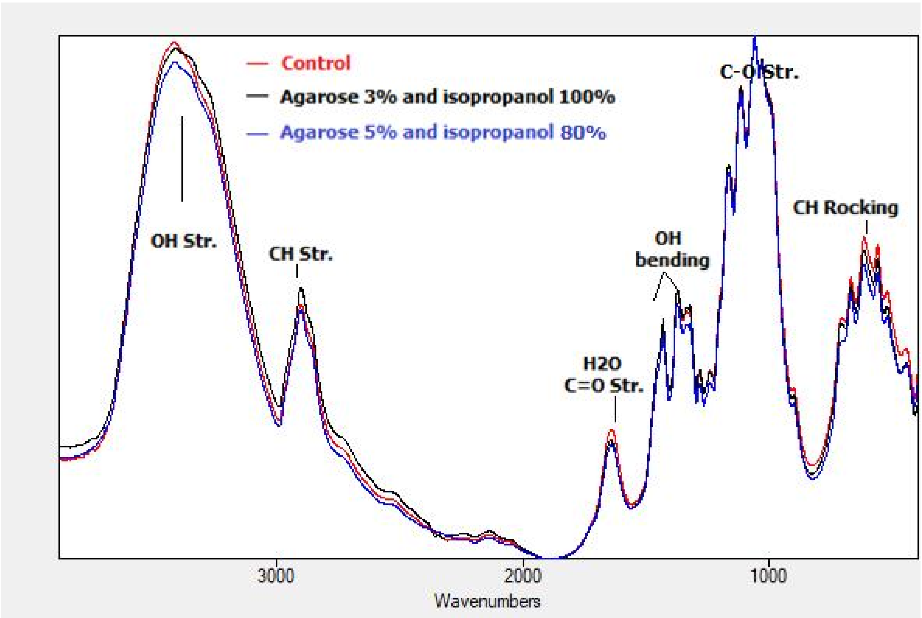

Fig. 19. FTIR spectra for agarose-based cleaning systems compared to the control sample.

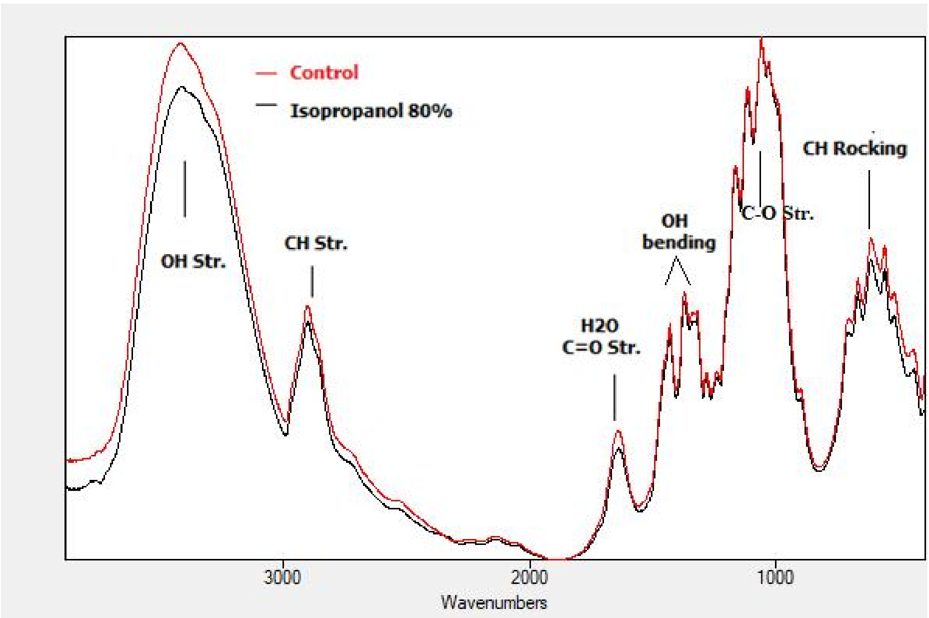

Fig. 20. FTIR spectrum for the sample treated with isopropyl alcohol 80% compared to the control sample.

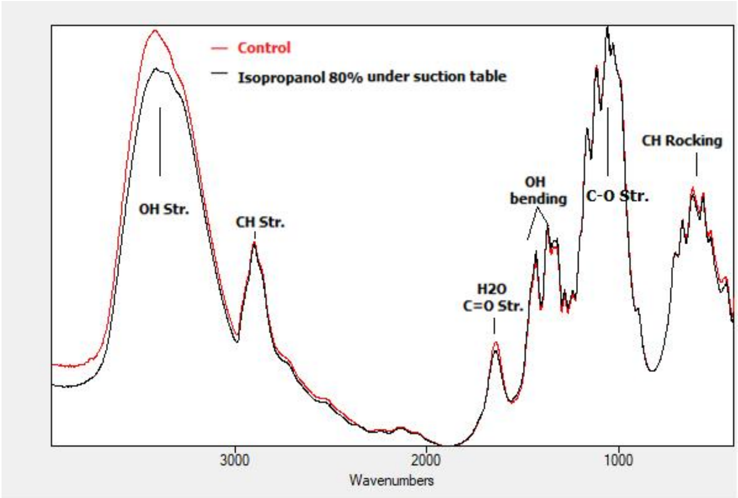

Fig. 21. FTIR spectrum for the sample treated with isopropyl alcohol 80% under suction table compared to the control sample.

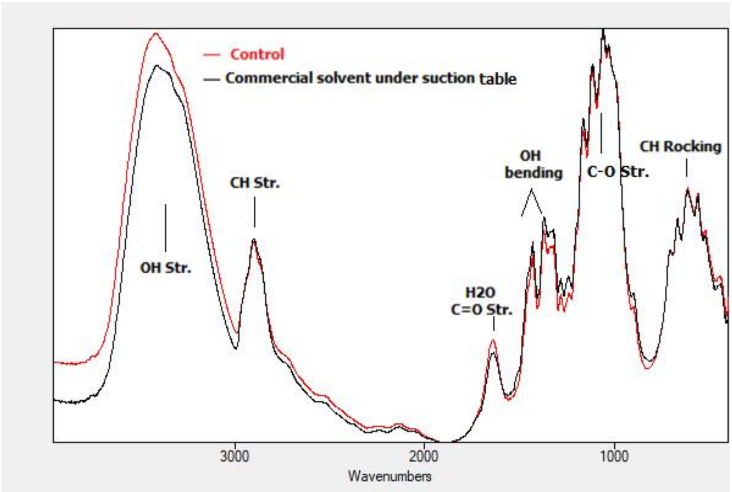

Fig. 22. FTIR spectrum for the sample treated with commercial solvent under suction table compared to the control sample.

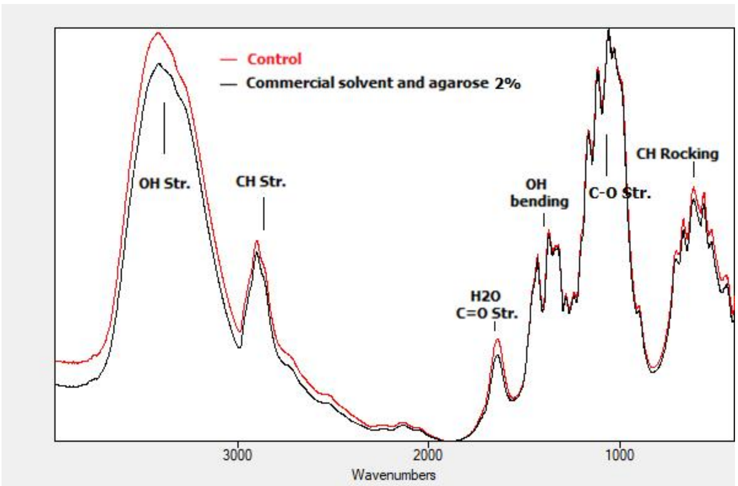

Fig. 23. FTIR spectrum for the sample treated with commercial solvent and agarose 2% compared to the control sample

Table 5: Functional groups for the samples with the aged pen ink stain post treatment compared to the control sample

Samples	Functional groups (cm^{-1})					
	OH stretching band	CH stretching band	H-O-H band + C=O stretching band	CH bending band + OH bending band	C-O stretching band	CH rocking band
Control sample	3416.18	2901.82	1644.19	1429.41-1355.92	1163.74-1028.09	615.46
Agarose 3% and isopropanol 100%	3414.36	2904.65	1647.04	1432.22-1353.09	1163.74-1030.91	615.46
Agarose 5% and isopropanol 80%	3416.18	2901.82	1647.01	1432.22-1350.27	1163.47-1030.91	615.46
Isopropanol 80%	3419.01	2901.82	1644.19	1435.05-1353.09	1163.47-1030.91	615.46
Isopropanol 80% under suction table	3413.36	2901.82	1641.36	1432.22-1355.92	1163.47-1030.91	615.46
Commercial solvent under suction table	3416.18	2901.82	1644.19	1435.05-1353.09	1163.47-1030.91	615.46
Commercial solvent and agarose 2%	3416.18	2901.82	1644.19	1432.22-1355.92	1163.47-1030.91	615.46

2.5. Mechanical testing: tensile breaking strength analysis

2.5.1. Unaged set-in pen ink stains

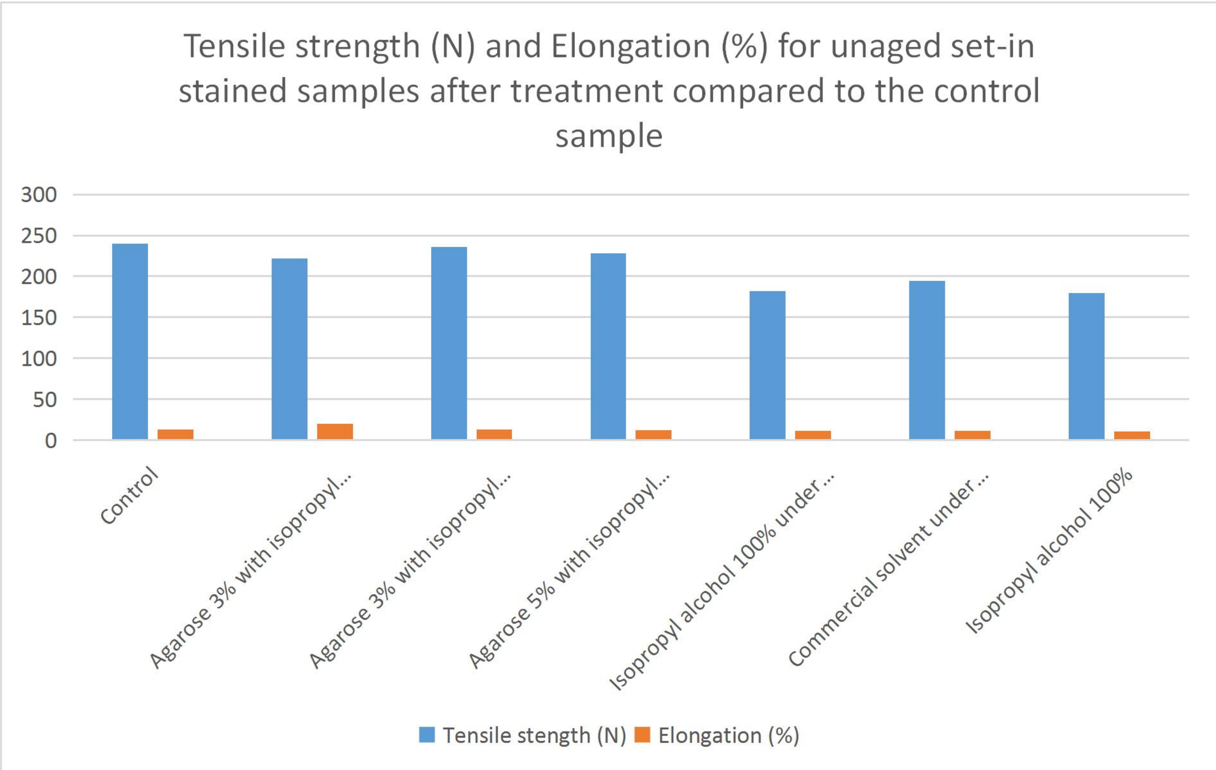

Fig. 24. The tensile strength and elongation for treated samples compared to the blank sample.

Table 6: Tensile strength and elongation for treated samples compared to the control sample

Samples	Tensile strength(N)	Elongation (%)
Control sample	239.9	13.34
Agarose 3% with Isopropyl alcohol 80%	211.6	19.97
Agarose 3% with Isopropyl alcohol 60%	236.3	13.00
Agarose 5 % with Isopropyl alcohol 60%	228.4	12.30
Isopropyl alcohol 100% under suction table	182.2	11.43
Commercial Solvent under suction table	194.2	11.17
Isopropyl alcohol 100%	180.00	11.00

Results revealed that treatment with 100% isopropyl alcohol, both with and without suction, as well as with the commercial solvent, resulted in a notable reduction in both tensile strength and elongation, compared to the control sample. This indicates that while these solvent systems are effective in stain removal, they may also compromise the mechanical integrity of the cotton substrate. The observed decrease is likely due to solvent-induced degradation of cellulose fibers, including possible chain scission, loss of hydrogen bonding, and reduction in fiber cohesion [47], particularly under conditions involving high solvent concentration or mechanical assistance from the suction table. In contrast, samples treated with agarose-based cleaning systems exhibited only minor changes in tensile strength and elongation when compared to the control. This highlights the gentler nature of gel-based treatments, which offer better control over solvent delivery and penetration, reducing the risk of fiber weakening. These findings are illustrated in (Fig. 24) and Table 6.

Samples	Tensile strength		Elongation	
	Value (N)	Change %	Value (%)	Change %
Control sample	239.9	--	13.34	--
Agarose 3% with Isopropyl alcohol 80%	211.6	- 11.79	19.97	+ 49.70
Agarose 3% with Isopropyl alcohol 60%	236.3	- 1.50	13.00	- 2.55
Agarose 5 % with Isopropyl alcohol 60%	228.4	- 4.79	12.30	- 7.79
Isopropyl alcohol 100% under suction table	182.2	- 24.05	11.43	- 14.31
Commercial Solvent under suction table	194.2	- 19.05	11.17	- 16.26
Isopropyl alcohol 100%	180.00	- 24.96	11.00	- 17.54

2.5.2. Aged pen ink stains

The samples treated with 3% agarose and 100% isopropyl alcohol showed only a slight reduction in tensile strength and elongation compared to the control, suggesting minimal impact on the mechanical integrity of the samples. In contrast, treatments involving 100% isopropyl alcohol, the commercial solvent (both applied directly and under suction), as well as agarose-based systems combined with isopropyl alcohol, resulted in a marked decrease in tensile strength relative to the control sample. These results, presented in (Fig. 25) and Table 7), indicate that while such treatments may enhance stain removal efficiency, they also pose a higher risk of fiber weakening and loss of structural stability—particularly in aged substrates where the cellulose may already be partially degraded.

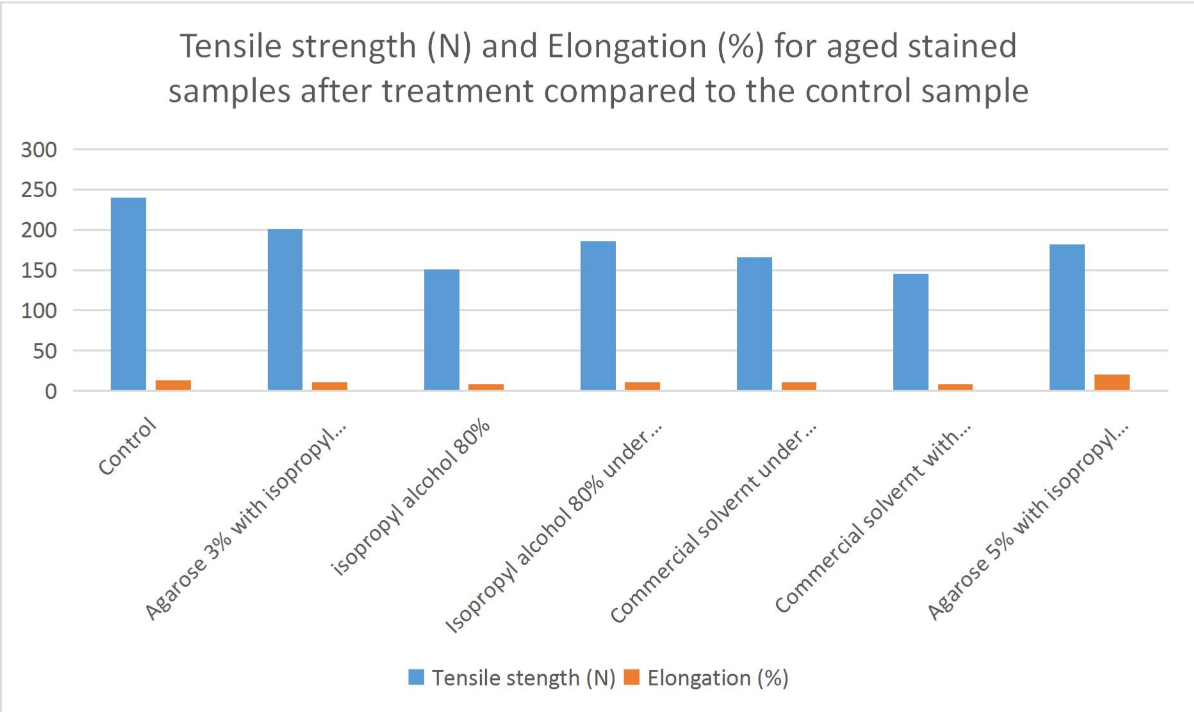

Fig. 25. The tensile strength and elongation for treated samples compared to the control sample.

Table 7: Tensile strength and elongation for treated samples compared to the control sample

Samples	Tensile strength(N)	Elongation (%)
Control sample	239.9	13.34
Agarose 3% with Isopropyl alcohol 100 %	201.3	10.99
Isopropyl alcohol 80%	151.2	8.40
Isopropyl alcohol 80% under suction table	185.7	10.80
Commercial Solvent under suction table	166.3	10.63
Commercial Solvent with Agarose 2%	145.7	8.27
Agarose 5% with Isopropyl alcohol 80%	181.9	20.03

3. Conclusion

This study investigated the effectiveness of agarose-based cleaning systems (i.e., agarose 3% with isopropyl alcohol 80%, agarose 3% with isopropyl alcohol 60%, and agarose 5 % with isopropyl alcohol 60%) compared to conventional solvent-based methods (i.e., isopropyl alcohol 100% and commercial solvent, both with and without suction assistance) for removing both unaged and aged pen ink stains from cotton textiles, while also assessing their impact on the fabric's chemical and mechanical integrity. The findings clearly demonstrate that mechanically assisted solvent applications, such as 100% isopropyl alcohol under suction, delivered the highest cleaning efficiency, evidenced by strong visual, colorimetric, and microscopic results. However, these treatments also caused the most significant reduction in tensile strength and elongation, indicating potential damage to the cotton textile substrate due to aggressive solvent action and mechanical stress. The samples treated with the commercial solvent under a suction table showed very good results in terms of stain removal, as confirmed by visual and microscopic examinations. However, this treatment was also associated with a noticeable decrease in tensile strength. On the other hand, agarose-based systems, particularly those composed of 3% agarose with 60% or 80% isopropyl alcohol, provided a balanced performance. These treatments offered moderate cleaning effectiveness with minimal impact on the color, the mechanical properties and chemical stability of the cotton fibers. The treatment with 5% agarose and 60% isopropyl alcohol resulted in low tensile strength, although visual inspection and colorimetric analysis indicated satisfactory cleaning performance. FTIR analysis confirmed that these gel-based systems induced only slight hydrolytic or oxidative changes, in contrast to the more pronounced alterations observed with direct solvent application, especially under suction. Additionally, for aged stains—which are typically more resistant to cleaning—effective outcomes were achieved using 80% isopropyl alcohol under suction and commercial solvents, especially when applied with agarose or suction support. Samples treated with 100% isopropyl alcohol yielded unsatisfactory outcomes, as evidenced by visual inspection, colorimetric data, and mechanical performance. These findings stress on the significance of tailoring the cleaning method to the type of stain and the condition of the substrate. Notably, combining agarose gels with moderate solvent concentrations allows for controlled delivery, reduced solvent penetration, and lower mechanical stress, which are essential for the preservation of historical textiles. Overall, this research confirms that rigid agarose gels are a promising, safe, and environmentally responsible alternative to conventional solvent systems in the conservation of ink-stained cotton fabrics. Future studies should continue optimizing gel compositions and exposure times to enhance cleaning efficacy while safeguarding artifact integrity.

4. Conflicts of interest

The authors confirm that there are no known conflicts of interest associated with this publication and there has been no significant financial support for this work that could have influenced its outcome.

5. Funding

(Not applicable)

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