

ART OF INSTANT BLACK INK FOR ETHIOPIC MANUSCRIPT RESTORATION

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Abstract

Traditional Ethiopian inks, primarily used in manuscripts and painting, have significant historical and cultural value. However, their production has gradually declined due to competition from fabricated inks. There is also a shortage of written recipes. In parallel, manuscript conservation faces mounting challenges, including ink loss and incompatibility with modern materials. This study aims to address the gap by researching the physical and chemical properties of traditional Ethiopian instant black ink. The methodology involved reconstructing traditional ink using a historical recipe. To ascertain the composition, stability, and authenticity of the produced inks, a range of chemical and physical analyses was conducted. The findings of this study indicate that instant black ink can be readily reproducible and possesses promising chemical and physical stability. This ensures the authenticity and sustainability of manuscript conservation. The study has implications for addressing ink fading in manuscripts by providing a model for integrating traditional materials with modern conservation practices.

Keywords: Authenticity; Manuscript; Instant ink; Restoration; Stability; Traditional

Introduction

Ethiopia, a nation with a rich and venerable history stretching back millennia, has a remarkable manuscript tradition. Ethiopic manuscript culture uses the Ge'ez script and has an uninterrupted long history [1]. This culture used parchment for the recording of religious, historical, and literary texts since antiquity [2]. The Ethiopian scribal practices played a pivotal role in the transmission of knowledge throughout the Horn of Africa. This tradition was intricately intertwined with the beliefs and practices of the Ethiopian Orthodox Church. The church has been instrumental in the production and preservation of these invaluable manuscripts [3]. In line with this, the scribes had the skill to produce mainly black and red inks by mixing different plant species and minerals [4].

Nevertheless, a significant number of manuscripts in the collections of churches, monasteries, and government institutions have deteriorated due to anthropogenic and natural factors [5]. One of such deteriorations is ink loss [6]. In this regard, field observation of the manuscripts from Lake Tana monasteries, the Institute of Ethiopian Studies, and the National Archives and Library Service revealed ink loss due to flaking, bleeding, erasure, and fading. For

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instance, the manuscripts including IES MS 3164 (31r), IES MS 439 (87r), IES MS 2184, IES MS 695 (1r), NALS 157 (192r) (Fig. 1a), NALS 285 (10r) (Fig. 1b), and NALS 860 (79v) have experienced ink loss.



Fig. 1. Ink loss a, NALS 157 (192r), and b, NALS 285 (10r)

Despite the extensive nature of the problem, efforts to restore ink loss and damaged manuscripts remain at their traditional level. Traditional restoration also started incorporating contemporary fabricated inks [7]. For instance, soot from burned paper, oil, and manganese are new additives used in ink-making for manuscript writing in Ethiopia [4]. These additions are against the principles of authenticity [8]. On the other hand, research into the characterisation of materials and the reconstruction of Ethiopian manuscript inks is in its infancy.

Some of the research focused on characterising the inks and pigments used in the Ethiopian manuscripts. The characterisation of Ethiopian inks and pigments used on the Ethiopian magic scrolls was carried out using Infrared Reflectography (IRR), spectroscopic analyses, and Ion Beam Analysis (IBA) [9]. In addition, the Raman Laser Microspectrometry analysis on two Ethiopian Miracles of Mary was conducted [10]. However, the studies' conclusions were limited to the consistency of composition with the existing literature and the foreign origin of non-carbon-based inks [9], [10].

Furthermore, the reconstruction of Ethiopian ink recipes is encumbered by two factors. These are the lack of precise measurement methods and the limitation of identifying chemical properties. Most recipes were transmitted from generation to generation orally [4]. Therefore, there is a limitation in the written recipes for ink production. Nevertheless, there are different recipes documented by Fekade Selassie Tefera and Sergew Hableselassie. Tefera discussed nine different recipes of black ink. Based on his interview with Tefera, Sergew also introduced six black-ink recipes [4], [11]. Sean Michael Winslow, consulting the previous two and contemporary practitioners, also introduced several recipe lists [12].

Although it is for painting, German iconographer Annegret Marx tried to reconstruct the recipes for ink [7]. However, none of those recipes indicated the chemical properties of the ingredients and the product. Besides, except for Marx's case, the recipes lack appropriate measurements for ingredient proportions.

Therefore, the main objective of this study was to reconstruct instant black ink in a laboratory using traditional ingredients for manuscript restoration. It was based on one of the recipes of Hable-Selassie and Tefera. This production of ink, using a suitably measured composition and known chemical and physical properties, has significant implications. It can be used as an input to reconstruct further existing recipes and provide traditional material for scientific analysis [13]. It can also provide authentic materials for the restoration of manuscripts.

This is because laboratory production enables consideration of colour matching, stability, uniformity, and compatibility [14].

Materials and Methods

Ink ingredients

The ingredients for Ethiopian Black instant ink were taken from the recipes of Tefera and Hable-Selassie [4], [11]. Tefera was a scribe and experienced in traditional ink preparation. In addition, Hableselassie interviewed many craftspersons, including Tefera. Accordingly, water, the leaf of redwing (*Pterolobium stellatum*), juice from roasted barley (*Hordeum vulgare*), and juice from the leaf of jimsonweed (*Datura stramonium*) were the ingredients (Fig. 2). Redwing is a tall, scrambling shrub with woody, rope-like stems and leaves that are compound with 7 to 15 pairs of leaflets. This plant is traditionally used for wound treatment. Scientific research also proved that the juice extracted from its leaves has antimicrobial potential [15]. The roasted and ground leaves of this plant are also an essential ingredient in four different recipes for black ink [4].

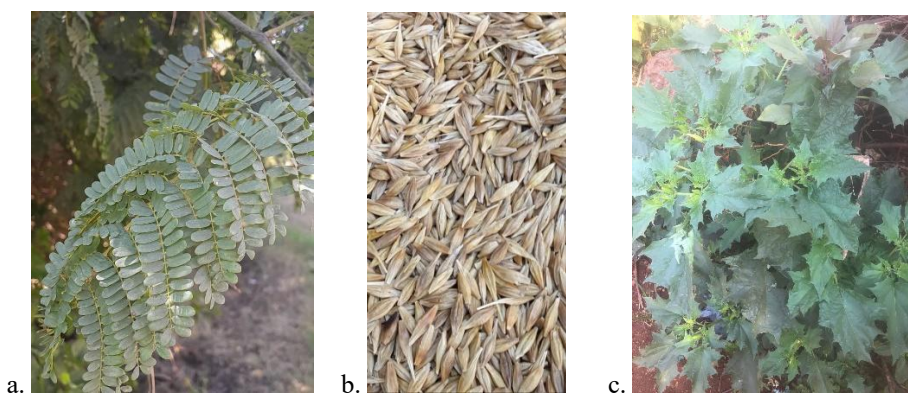


Fig. 2. a: Redwing; b: Barley; and c: Jimsonweed

Barley is the fifth most crucial traditional cereal in Ethiopia [16]. This crop has been used as food to treat diseases such as gastritis and heartburn, and the straw is used as animal feed and for thatching roofs [17]. It is also used in the preparation of traditional inks, replacing wheat (*Triticum aestivum*) and finger millet (*Eleusine coracana*) [11]. Jimsonweed was also one of the ingredients for this ink production. It is a plant with a higher traditional medicinal value in the country. Its leaf extracts are helpful in the treatment of leukoderma, dermal disorders, ulcers, bronchitis, jaundice, hysteria, insanity, heart disease, fever, and piles [18], [19].

Preparation process

The first step was collecting the ingredients. The leaves of redwing were collected from the garden at Bahir Dar, Ethiopia. The fresh leaf was roasted until it turned charcoal black. The charcoal-black leaf was then ground into a fine powder (Fig. 3a). The barley was bought from farmers around Bahir Dar. The dry 250 grams of barley were roasted in a pan. The roasted barley was boiled again using 600 mL of distilled water for 20 minutes. It was then left for cooling. After that, the liquid was carefully poured into a jar using a strainer (Fig. 3b). The collected jimsonweed leaves were juiced utilizing a juicer. The initial preparation of the ingredients was made at home.

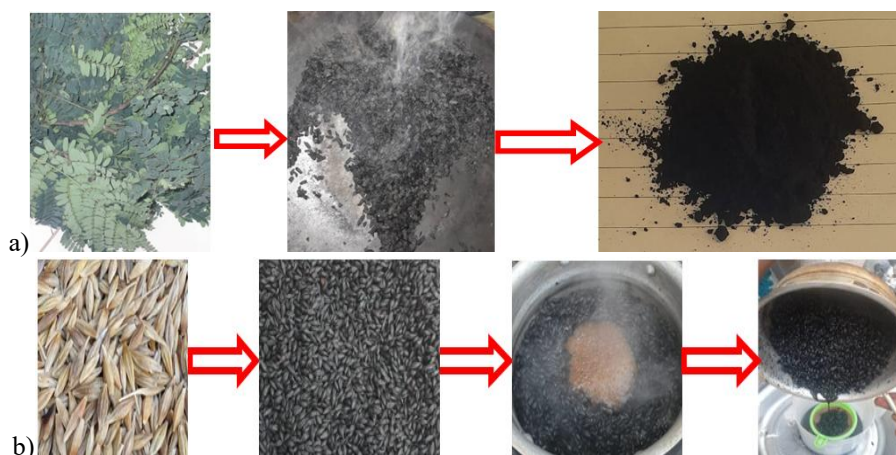


Fig. 3. a) Preparation process of the powdered leaf of roasted redwing;
 b) Preparation process of the extracted solution from roasted barley

Later, different ingredient combinations were tested in the lab to make ink. Four different ink samples were prepared, each with a different ingredient ratio (Table 1). This was done to achieve an appropriate ingredient ratio based on their chemical and physical properties. Samples were labelled as instant black ink (IBI) one to four. The first sample (IBI1) comprised 81.08% of solution from roasted barley (*Hordeum vulgare*) (HV), 10.81% of juice from the leaf of jimsonweed (*Datura stramonium*) (DS), and 8.10% of redwing (*Pterolobium stellatum*) (PS). The second (IBI2) includes 88.26% of HV and equal 5.88% of the ratio of DS and PS. The ratio of 66.67% HV, 20% of DS, and 13.33% of PS was used in IBI3. In IBI4, 62.5% of HV, 18.75% of DS, and 18.75% of PS were used (Fig. 4). A higher HV ratio was tested in IBI1 and 2, whereas in IBI3 and 4, the DS and PS ratios increased significantly. An equal ratio of all ingredients turned the inks green during preliminary preparation, which indicates the need to use a smaller ratio of DS plant.

Table 1. Samples with different ingredient ratios

INGREDIENTS	UNIT AND PERCENTAGE	IBI1	IBI2	IBI3	IBI4
HV	MI	30	30	20	20
	Percent	81.08%	88.26%	66.67%	62.5%
DS	MI	4	2	6	6
	Percent	10.81%	5.88%	20%	18.75%
PS	gram	3	2	4	6
	Percent	8.10%	5.88%	13.33%	18.75%



Fig. 4. Ingredients and their measurements of IBI4

The prepared inks were tested by writing on pieces of parchment made from goat skin (Fig. 5). The parchment was prepared without liming. In accordance with practices in traditional Ethiopian book craft, parchment was prepared without the use of lime [12]. Then the skin was cut into 50×50mm pieces. The Ethiopic letter “ገ” was written on each piece using the same traditional ink pen (Fig. 5). Attempts were made to write the same-sized letters across all pieces. In addition, paper was used to assess feathering and ghosting. This is because paper is more porous than parchment, which makes it easier to analyse feathering and ghosting (or bleed-through) [20].

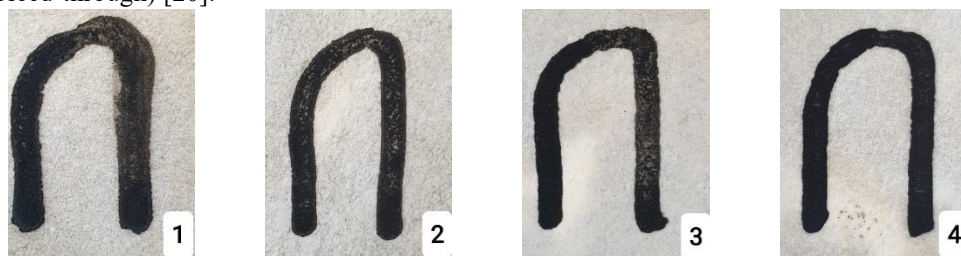


Fig. 5. Pieces of parchment with a letter written by produced ink

Methods of analysis

Chemical and Physical properties of the inks

The pH measurement was undertaken using an AD800 Adwa PH meter. It is used to measure the concentration of hydrogen ions $[H^+]$. The concentration of hydrogen ions is used to measure the acidity or alkalinity. PH has been determined experimentally and then converted to pH values using mathematical relations [21]. In this study, it was used to understand the acidity and alkalinity of ink solutions. Besides, the TDS and EC of the solutions were measured using a Portable digital EC and TDS meter. It was done to measure electrical conductivity and determine the amount of dissolved solids.

The viscosity of the inks was also measured using the VISCO STAR plus Rotary viscometer. Viscosity is a property of all liquids manifested by internal resistance to flow or shear. It is also a drag force and a measure of the fluid's frictional properties [22]. This was done by immersing spindle four into each sample solution at 50 RPM. All measurements of pH, EC, TDS, and viscosity were taken at 24°C. The drying time of ink samples was measured after writing on parchment.

Table 2. Chemical and physical properties of the sample solutions

No of sample	Amount in ml	PH	TDS	EC	viscosity	Drying time/minutes
IBI1	37ml	6.9	1650ppm	3300 μ S/cm	3 cP	13
IBI2	34ml	6.84	1697ppm	3522 μ S/cm	3 cP	9
IBI3	30ml	7.22	1805ppm	3788 μ S/cm	4 cP	5
IBI4	32ml	7.15	1640ppm	3280 μ S/cm	5 cP	2

Visual observation

Observation using a Dino light microscope, a microscope camera, and the naked eye was made on the written ink. It was made to identify colour uniformity (evenness and distribution), feathering, and bleed-through.

Results and Discussion

Chemical and physical properties

The pH of the sample inks increases from acidic to alkaline as the amount of roasted leaf powder increases. The added PS powder increased the solution pH from 6.9 in IBI1 to 7.15 in IBI4. The only difference is IBI3, where the PH rises to 7.22. In short, the first two have acidic characters, and the remaining two are alkaline (Fig. 6). But the alkalinity is decreased in IBI4.

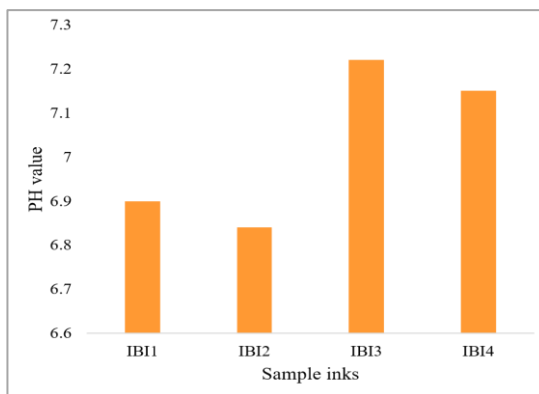


Fig. 6. pH of sample ink solutions

Research suggests that the pH of leaves is more often acidic. For instance, the ground leaf pH was higher than the non-grounded one, according to [23]. Similarly, the juice from roasted HV and the juice from the leaf of DS were 5.23 and 6.4, respectively. However, applying heat or roasting decreases the acidity of leaves [24]. As a result, the alkalinity of the roasted PS flour is consistent with the existing literature.

The PH of the writing support is expected to be neutral, as the PH (acidity and alkalinity) of the inks affects the media's stability. Parchment is a post-mortem tissue composed mainly of collagen. It is the extended network of hydrogen bonding exerted both among amino acids and between water molecules. Thus, the acidic pH can cause hydrolysis and oxidation, which may reduce the size of collagen fibrils [25]. In this regard, iron gall ink, which was used across Europe during the Middle Ages, is known for its tendency to cause significant degradation. The gradual development of acidic pH and the hydrolytic formation of gallic acid from tannins were recognized as the causes of deterioration of writing supports [25]. Therefore, the IBI3 and IBI4 are closer to ideal inks, which have alkaline properties. The IBI4 is more appropriate because it has a relatively neutral property than the IBI3.

Electrical conductivity (EC) is a measure of the ability of a conductor to carry an electric current. In an aqueous solution, EC is defined as the total current-carrying capacity of all ions. This property is contingent upon the number of ions present within a unit volume of solution, in addition to the mobilities with which each ion is capable of moving in response to the applied electrical potential [26]. Total dissolved solids (TDS) were also measured as part of characterization. TDS is the total concentration of inorganic and organic matter in an aqueous solution. It comprised ions such as calcium, magnesium, sodium, bicarbonate, chloride, sulfate, and other organic components [27]. The higher concentration of these ions influences the stability of parchment. They cause degradation through oxidation and gelatinization [25].

The EC and TDS of sample ink solutions increased gradually from IBI1 to IBI3 and decreased at IBI4 (Fig. 7). A higher percentage of DS affects both the EC and TDS of the solutions. The higher EC and TDS were recorded, with the highest percentage (20%) for this plant in IBI3. Although it depends on several factors, TDS and EC follow a similar pattern in a solution: the more dissolved minerals, the more conductivity [28]. Similarly, in the sample inks, the relation between EC and TDS is parallel (Fig. 8). However, the slight decrease in IBI4 results in better control of the impact of inks on parchment stability.

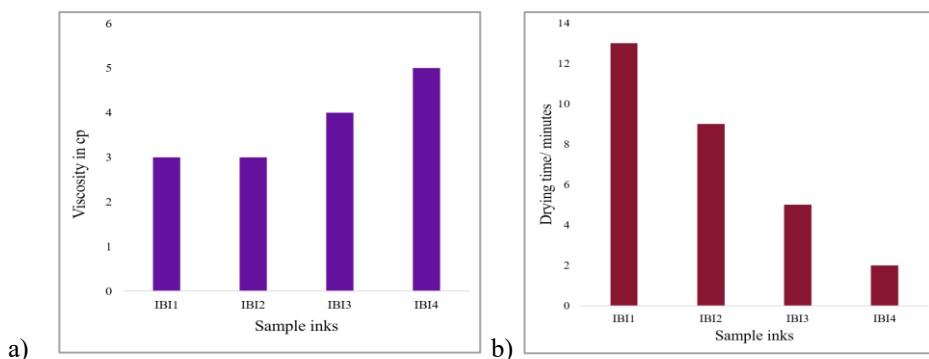


Fig. 7. a) Viscosity of sample ink; b) Drying time of ink solutions

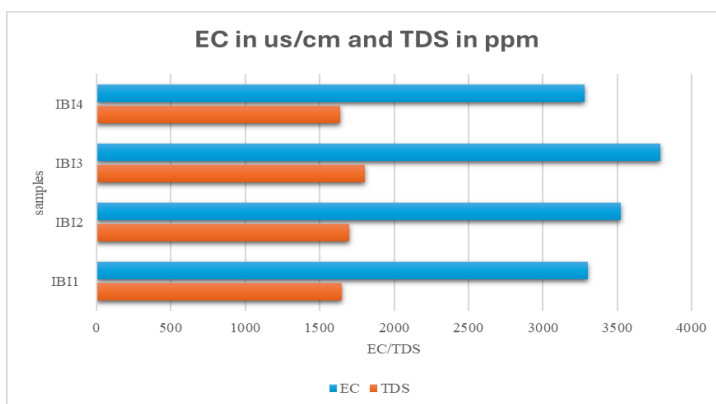


Fig. 8. EC and TDS of sample inks

The viscosity of the ink solutions increased steadily from IBI1 to IBI4 (Fig. 7a). The first two combinations have similar viscosity. But it increases in the IBI3 and IBI4 samples. It is due to the increase in the amount of powder of the roasted leaf of PS and the juice of DS. The viscosity of solutions can be affected by volume, concentration, temperature, particle size, shear rate, morphology, and density [29]. In this case, concentration significantly affects ink viscosity. A concomitant rise in its viscosity accompanied an increase in the concentration of the powder of PS. In addition, the relatively viscous nature is essential for ink applications, as it resists feathering and bleeding [30]. However, a higher concentration of powder in the absence of binders could cause flaking, as the failure of the ink's binders is one such factor [31].

The ink solutions were immediately measured for drying time upon application to the parchment pieces. The drying time of these samples decreased from IBI1 to IBI4 (Fig. 7b). IBI1 took the longest time to dry. Conversely, the IBI4 and IBI3 dried faster than the remaining two.

This is because of the increase in the powder content of the roasted PS leaf and the decreased solution percentage from HV. A faster drying time is recommended for writing inks on parchment. Parchment is a hygroscopic material; thus, a longer drying time will affect the stability [32].

In this study, it was possible to witness that the increase in the concentration of roasted and powdered leaf of PS resulted in a better drying time. Indeed, moisture content, size, surface area, shape, material ratio, particle size distribution, temperature, relative humidity, air velocity, and direct sunlight are also factors influencing drying time [33]. Nevertheless, the majority of previous variants demonstrated comparable properties across all samples, except for concentration. Furthermore, the drying time of the sample ink solutions was found to be inversely proportional to their viscosity. Drying time decreases with increasing viscosity.

Visual observation

Visual observation on the pieces that were used as a writing medium was undertaken to evaluate color uniformity, feathering, and ghosting. These parameters are observed on both the paper and the pieces of parchment. Color matching and uniform spread are among the criteria for selecting ink [14]. Although the inks appear uniform to the naked eye, none of the samples is evenly distributed when viewed under the Dino Lite microscope at 86 \times (Fig. 9).

In comparison, the ink is more evenly coated in the IBI4. IBI1 is less uniform, followed by IBI3. The gradual increase in PS concentration from IBI1 to IBI4 affects uniformity. A relatively smaller PS ratio in IBI3 results in a less uniform color. In addition, the sample inks are more uniform in parchment than in paper. In the pieces of paper, the different sample solutions indicated flaking after drying. This difference in reaction is due to the variety of surfaces. The various surfaces allow penetration of specific types of ink while deterring others [14].

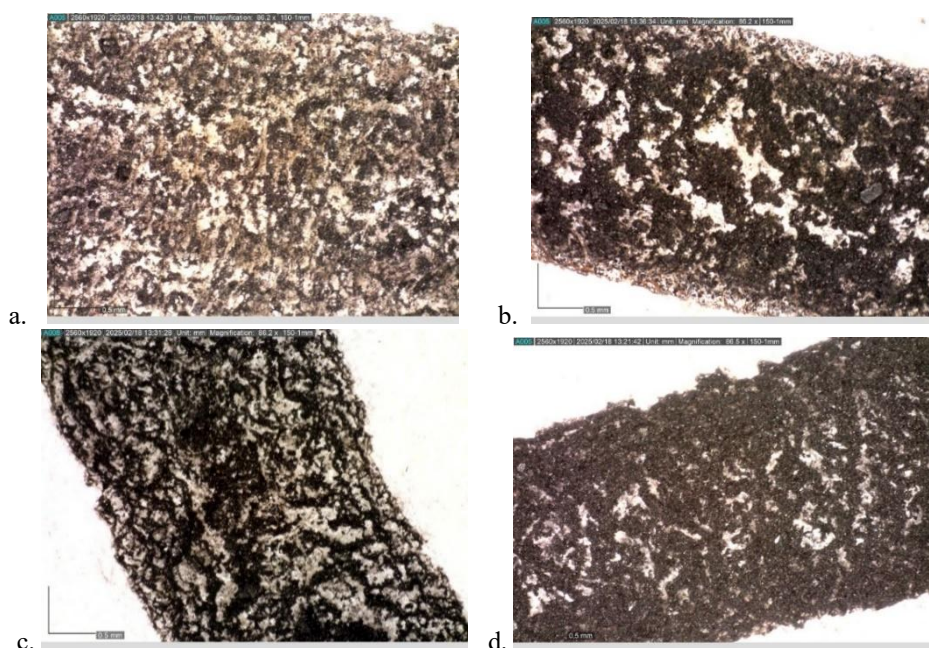


Fig. 9. Microscopic view of parchment pieces at 86 \times : A. IBI1; B. IBI2; IBI3; and D. IBI4

Another feature examined in the samples was feathering, defined as the spreading of ink that results in blurred, indistinct lines on the writing surface. This phenomenon can be attributed

to the surface's physical characteristics, the writing implement used, and the ink's properties [34]. This feature was more visible on paper than on parchment. The phenomenon may be attributable to the difference in porosity between parchment and paper. As a result, on the paper, feathering is more visible in the IBI1 sample and decreases in the 4th sample. But all the samples on the paper have feathering. Whereas, on the parchment, feathering is visible only on IBI1. The ink coverage of sample IBI1 is 1 mm wider than the other samples. It is 4mm as compared to 3mm of IBI2-4. In the remaining three, this is almost non-existent.

In addition, the ink on the paper has flaked, though the extent varies depending on the composition of the ingredients. Flaking was more clearly discernible under the microscope, particularly in areas not used for ink testing (Fig. 10). Accordingly, IBI3 shows more ink flaking among the samples. In this sample, the flaking is also more pronounced in the text part. IBI1 and IBI2 follow it. IBI4 shows better ingredient adhesion, likely due to PS. In line with this, the recipe from Tefera recommends adding boiled PS leaf to prevent ink from flaking off the parchment surface [4].

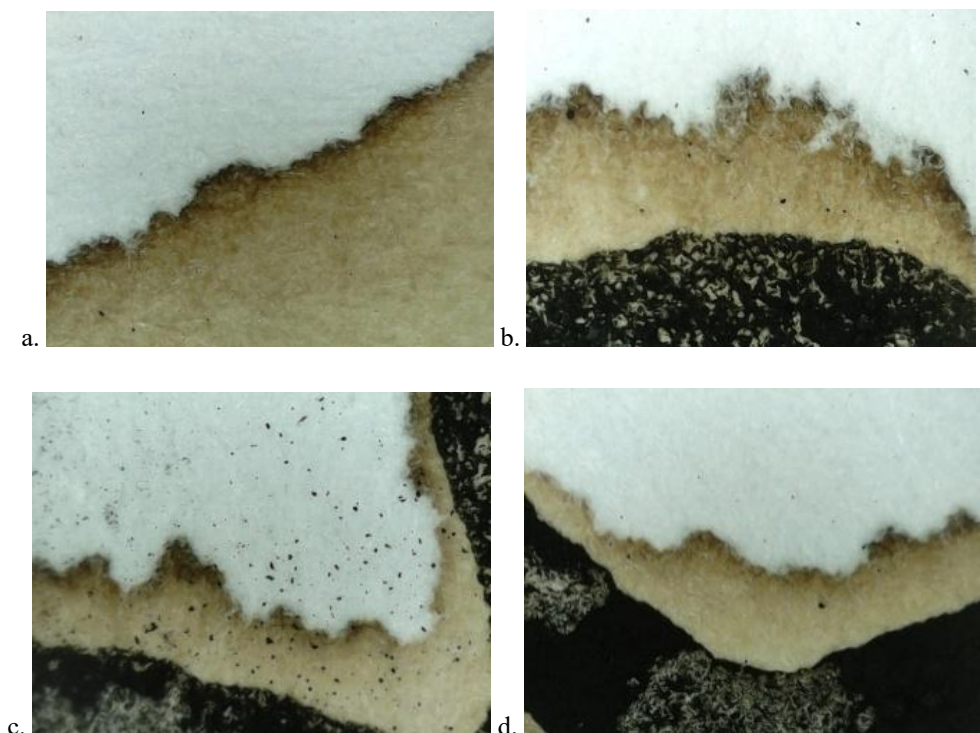


Fig. 10. Ink feathering on paper, left to right, IBI 1 to 4

The other important feature of ink observed in this study was ink bleed at the opposite part of the writing. This feature is more visible on paper than on parchment pieces. All the samples tested on paper, except IBI4, have experienced severe bleed-through. IBI4 has shown a slight difference and is better. Although it is not as severe as the paper, the parchment also showed bleed-through under a microscope camera (Fig. 11). Again, IBI4 is better than the remaining three. The second has more visible ink bleeds than the rest. Bleed-through is also more visible on the hair follicles than in the area without follicles.

The analyzed physical and chemical properties indicated that IBI4 is better than the other samples (Fig. 12). It is better in terms of limited ghosting and feathering. In addition, it has a relatively neutral pH, good viscosity, and ink stability. However, ghosting and feathering may be caused by a lack of binders, as binders can help prevent feathering, spreading, and bleeding [35].

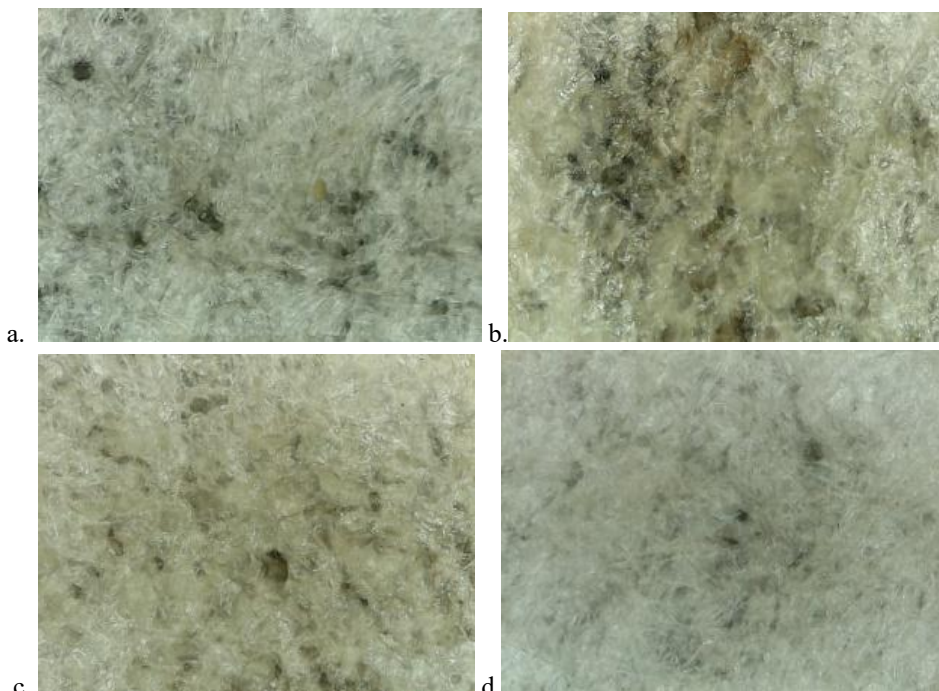


Fig. 11. Ink bleed-through on parchment, left to right, IBI1 to 4

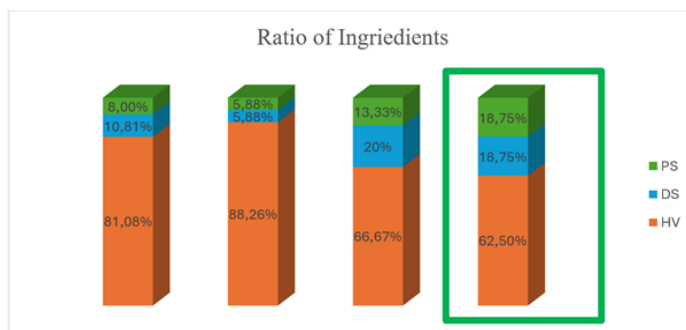


Fig. 12. Ratio of ingredients

As the manuscripts are written with classical inks, the restoration of ink loss needs a combined approach incorporating traditional materials and modern technology [36]. Traditional materials are important sources of promoting authenticity and sustainability [37]. In this regard, inks like IBI4 can be improved and adapted for retouching of ink loss. Additionally, this reconstruction of traditional inks provides an approach for conserving the craft of ink-making

through lab-based documentation. The craft is increasingly endangered due to the growing availability of accessible and inexpensive substitutes [38]. Therefore, such reconstruction provides an input for the survival of the craft and promotes the integration of traditional culture with contemporary technologies [39].

Conclusion

This research focused on the reconstruction of traditional ink for manuscript restoration. Instant black ink was made from traditional materials and subjected to physical and chemical stability tests. One of the samples tested (IBI4) has yielded promising results. This indicates the role of recipe reconstruction for preserving authenticity and maintaining sustainability in the conservation of documentary heritage. It also implies the importance of integrating indigenous wisdom with contemporary scientific knowledge and skill. However, limitations remained visible, particularly in uniformity/ evenness and slight ghosting. The ink's physical properties were not fully characterised. The absence of binders in the recipe was noted, which could affect the longevity and stability of the ink on the writing support. Above all, the inks' reactions to environmental variables over time were not considered in this study. Future research should address these limitations, in particular by improving the consistency of the ink and exploring the incorporation of binders to enhance its applicability for long-term manuscript conservation.

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