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Improving access: Multi-analytical survey of the Norwegian Sea trade archive to establish storage conditions, digitisation priorities and conservation treatments

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ABSTRACT

The Norwegian Sea Trade Archive (NST Archive) from the University of Bergen Library, Norway, is part of the UNESCO documentary heritage and contains unique documentation of the activity of companies that traded in stock fish in Norway and Europe (16th-20th century). The archive consists primarily of bound paper manuscripts written mostly in brown ink. We have surveyed this collection using a multi-analytical approach focused on the material characterization and condition of the paper carrier and inks. Our main aim was to support decision-making on establishing storage conditions, digitisation priorities, and type and extent of conservation treatments necessary to make the collection accessible.

After a first visual examination to detect and quantify damages, for the paper support analysis we measured surface pH, paper thickness and water absorbency. Additionally, on a quarter of all tested materials, we performed a SurveNIR analysis, providing information regarding paper pulp type, lignin, protein and rosin content, degree of polymerization, and mechanical properties. The archival material was grouped into three periods characterising the three milestones in paper production: handmade gelatine-sized rag papers, machine-made rag papers with gelatine and rosin sizing, and papers made from processed wood pulp.

Regarding the manuscripts' inks, we used iron and copper tests to determine the presence of Fe^2, Cu^{2+} and Cu^+ions, respectively. For the visual characterization and condition rating of the inks, we used the protocols described by the Netherlands Institute for Cultural Heritage. The variety of ink colours and thickness of the inked lines encountered in each document was noted down. Thus specific tendencies present in each historical period became apparent. Results of the solubility of ink in water and alcohol significantly narrowed treatment options.

General tendencies of paper and ink degradation based on differences in papermaking were observed. Surface pH measurement, SurveNIR pH, rosin and protein concentration data showed that most rag papers were of poor to fair quality (pH $\leq$ 6.0 and low protein content ($<$ 5 %)). Rosin-sized papers dated after 1840 had a lower pH (from 3.3 $\pm$ 0.2 to 5.0 $\pm$ 0.2) compared to gelatine-sized ones. Based on the obtained results, the overall condition of all the papers is good – slightly damaged but stable. The inks are not severely corroded, and there is no loss of text, although 20 % of the collection does show signs of iron-gall ink (IGI) corrosion. A limited number of documents, mostly before 1800, would require local antioxidative treatment. Due to the low pH of all the papers and the detection of ink corrosion, the collection should be stored in a cool environment, with low relative humidity.

After the survey, digitisation priorities were also established.

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1. Introduction

The Norwegian Sea Trade Archive from the University of Bergen Library, Norway, is part of the UNESCO documentary heritage and contains unique documentation of the activity of private companies that traded in stock fish in Norway and Europe (16th-20th century). Most of the archive originates from six large companies (Mowinkel, Krohn, von Tangen, Harmens, Mohn and Bugge) [1]. Some documents from the earlier period arrived at the Library in the 19th century, but the majority of the archive was donated in the 1930s and 1940s when the trade was finally closed. The collection includes accounting books, legal manuscripts, ship logbooks and correspondence, comprising ca. 90 shelf meters of bound and unbound material. From 1360, Bergen became an international trade centre when the Hanseatic merchants established themselves in the city. They introduced German accounting methods that functioned uninterrupted into modern times. The trade was based on the barter system. Fishermen delivered the dry cod to the merchants and received in exchange other merchandise such as grain, textiles, tools, and tobacco ([2] p. 225). Due to low prices set for fish, fishermen were often in debt. Accounting books recorded those debts, and these records became part of the capital that could be sold with the business ([3] p.22).

Hanseatic firms built their long, narrow company houses along the German Quay or Tysk Bryggen. Fish was stored on the ground floor, while offices were upstairs on the first floor. The whole building was not heated for fear of fire. Conditions were cold, humid, and frequently dusty, and all organic materials such as wood and paper absorbed the smell of dried fish. Some companies had better cleaning routines than others but overall, the whole archive had a similar history and was kept in similar conditions. In the University of Bergen Library, the archive was previously stored together; now it is divided into two rooms but both have similar environmental conditions due to climate control, introduced 15 years ago.

Surveys of collections are often implemented as part of a collection management strategy [4] for value and risk assessment, contributing to a larger preservation picture [5,6]. When a survey is performed for the purpose of digitisation, the collection has to be divided into two parts: those objects that are stable, safe to handle and digitise, and those that would require further treatment.

In the present study, the NST collection was surveyed in order to group materials by characteristic features and define treatment priorities and types of treatment, so that this archive could be made accessible in the reading room to researchers and through digitisation.

Access to bound archival materials is often dependent on the condition of the bindings. The survey of the bindings in the NST archive was previously undertaken [15]. The present survey concentrated primarily on the study of the paper support, its composition, and the extent of IGI corrosion, using a multi-analytical approach. Around 6 % or 133 objects out of a total of 2264 were randomly selected. The selection was organised by periods:

- earliest period – 1550–1799 - containing gelatine-sized rag papers, the most valuable items and least numerous; (14 items or 14 % of that period)
- middle period – 1800–1869 – containing documents when the paper started to change from handmade to machine-made and alum-rosin sizing was introduced; (45 items or 7 % of that period)
- third period – 1870–1930 – containing the most recent and most abundant documents, when rosin-sized, chemically bleached pulp became the predominant paper type. (74 items or 5 % of that period).

After a first visual examination to evaluate the presence of evident damages, a more thorough investigation of the collection was required, as the assessment of pH value and mechanical strength or degree of polymerization (DP) of paper artefacts necessitates the use of specialized instruments and/or material sampling techniques [8]. SurveNIR survey tool [9,10] was selected as it allows for non-destructive analysis of paper-based collections, defining the types of paper pulp (rag, bleached chemical pulp, mechanical woodpulp), sizing (protein, rosin) and estimating fibre integrity (tensile strength and DP). Recent collection surveys of both paper and photographs often utilise SurveNIR to define their condition and preservation strategies [11–13].

To evaluate the risk of acid hydrolysis, one of the main factors in the deterioration of paper-based collections [16], surface pH was measured on all the samples. One-third of the selected samples were analysed with SurveNIR [11]. Also, the thickness of the paper and the hardness of the size were determined, since it has been suggested that thicker paper and harder size could provide partial protection to the paper fibres from ink corrosion [19,14].

Regarding inks, since the main manuscript ink in most of the Western archives is IGI, the systematic survey categories introduced by the Canadian Conservation Institute (CCI) were adopted here as a standard for risk assessment of manuscript inks [14].

Registration of ink colour and ink thickness was also considered, as it has been suggested by Season Tse et al. [14] that darker and thicker ink usually leads to greater damage to the paper support. Much research has been done on the study of the causes of ink corrosion and hydrolysis [17–19]. Ferrous (Fe^{2+}) ions promote the Fenton reaction and formation of hydroxyl radicals and lead to oxidative degradation [20]. At the Netherlands Institute for Cultural Heritage, a test with bathophenantroline indicator paper was developed to identify the presence of ferrous ions on inks, along with a protocol for analysing the condition of IGI [21–23], which we followed here. Other transition metal ions, especially copper, present in IGI can also contribute to oxidation processes [20,24]. The presence of copper was determined with Cuprotesmo paper [25]. To determine the solvent to be used for potential future conservation treatments, the manuscript inks, including demarcating lines and marginalia, were tested in various concentrations of water and isopropyl alcohol.

2. Research aim

The overarching aim of this study is to provide a basis for recommendations of storage conditions, and the extent and type of conservation treatment necessary to secure access to the collection. To achieve this goal a multi-analytical survey of the Norwegian Sea Trade Archive from the University of Bergen Library was undertaken to determine the condition of the paper support and manuscript inks.

3. Experimental

3.1. Paper support

3.1.1. Visual examination for damages

Each object was surveyed and the most prominent damages that affect the stability of the paper support and limit the access were noted down in an Excel sheet. Among those are:

- Obvious signs of microbial attack
- Water damage, tide lines
- Superficial and engrained dirt
- Tears
- Fire damage
- Foxing that might indicate the presence of metal particles and/or mould

- Yellowing and acid burn
- Pressure sensitive tape
- Metal fasteners

3.1.2. Paper thickness

Paper thickness was measured with a mechanical Cordix calliper that has a minimal measurement point of 0.1 ± 0.05 mm. Each sheet of paper was measured individually at the bottom of the sheet. Three folios were measured in each bound volume once. In the case of thin papers, it was easy to see the position of the arrow on the mechanical calliper if it is between 0 and 0.1 to determine that it is <0.1 mm. Thus, papers were divided into three categories: thin papers (<0.1 mm), medium thick papers (0.1–0.2 mm) and thick papers (>0.2 mm).

3.1.3. Water absorbency

Water absorbency was determined by placing a drop of deionised water (0.05 ml), always using the same pipette, on a clean section of paper, as described in ([26], p.167). If water was absorbed in less than 30 s, it was considered slack-sized; if it took between 30 s and 1 min, it was placed in the category of medium-sized papers; and if in over 1 min there was no sign of absorption, the paper was determined to be hard-sized. In some documents, there were signs of water damage. In those cases, water absorbency was measured in two areas: clean and unaffected, and within a tideline for comparison.

3.1.4. Surface pH

Surface pH was measured according to the TAPPI T529 standard [27] and following a more refined description from the CCI guidelines [28]. A piece of blotter was placed under the paper to be tested and a drop of deionized water (0.05 ml) was placed on an area of clean paper not affected by mould or water damage; a flat electrode was placed over the drop making sure that there is a layer of water between the electrode and the paper. TAPPI T 529 standard [27] does not specify the size of a drop, but CCI guidelines indicate that it can vary between 50 and 100 µL (0.05 - 0.1 ml) per drop [28]. Portable Amtast AMT28 with a flat surface electrode was used. To avoid damage to paper only one measurement was done for most of the objects. To estimate the average standard deviation for the pH value measurements, seven items were measured four times each.

3.1.5. SurveNIR

As an additional tool, SurveNIR analysis was performed on a third of all tested materials. The analysis was performed in situ together with Dirk Lichtblau from Lichtblau e.K. in Dresden, Germany as the provider of SurveNIR. Its spectrometer with an InGaAs diode detector (512 pixels) measures NIR-spectra in the wavelength range of 1100 to 2200 nm at an approx. 2 nm resolution [11]. The evaluation of SurveNIR results is based on chemometric analysis. An extensive database of previously measured paper samples has been used to develop the numerous chemometrical models, which are modularly embedded in the SurveNIR software SUSO. These chemometric models evaluate the measured NIR-Spectra to determine the paper pulp type (rag, groundwood or chemical pulp) as well as relevant parameters to describe the paper like pH, DP, protein lignin and rosin content as well as mechanical properties e.g., tensile strength.

Each result is an average of eight NIR-spectra measurements taken from the same spot on a page. The position of the book was not changed during measurement. The program automatically filters and averages the measured NIR spectra and evaluates the results providing instantaneous information. In the described survey, all papers were measured in the middle of the page, not affected by water damage or other forms of degradation. Errors of determination were taken from [11].

3.1.6. Paper pulp type

Microscopic examination of paper samples was undertaken to confirm the identification of paper pulp type by SurveNIR. 19 manuscripts were analysed in total. Paper fibres were collected with tweezers and stained with the following solutions: phloroglucinol with HCl (to identify lignin), Hertzberg solution (to distinguish between chemical, mechanical and rag pulps), and Lofton-Merritt solution (to distinguish between unbleached and bleached chemical pulps and semi-chemical pulps). The samples were observed under an optical microscope Zeis Axioplan 2 Imaging system (with tungsten halogen lamp, HAL 100) in bright field, and registered using a Nikon Digital Sight 10 camera with control software NIS-Elements Basic Research. The preparation of stain solutions and interpretation of results was based on the literature and standards (phloroglucinol ([29], p. 66), Hertzberg [30], Lofton-Merritt [31]).

3.2. Manuscript inks analysis

3.2.1. Examination under visible and UV light

Manuscript inks were examined visually for signs of corrosion under visible and UV lights, following the guidelines outlined by Birgit Reissland [22,23]. A UV-A lamp with magnifying lens was used (368 nm/16 W) for this examination.

The line thickness was measured with a transparent ruler (1 mm dividers) observed in two ways: as the general thickness of the line, and as the amount of extra thick lines present (as in the case of decorative elements in initials and baroque curvatures). Based on Season Tse et al. recommendations [14], the information was gathered in four categories: extra thick (>1.5 mm), thick (1.0–1.5 mm), medium thick (1.0–0.5 mm) and thin (<0.05 mm). The amount of extra thick lines and ink blots was noted down in the spreadsheet as being of four types: 0 %, <10 %, >10 %, and ca. 25 % compared with the rest of the manuscript ink.

Considering the need to systematise the colour observation, and since in most archival settings there is no access to colourimeters, we have used paint swatches from the paints industry – NCS (Natural Colour System) [32], as colour references. NCS colour scan routinely employed in the paint industry is used also in wallpaper conservation [33], and colour swatches are part of the documentation of personal paper conservation practice to determine the paper colour before and after treatment, but this method has not been published yet. The ink colours were grouped into five categories: very dark (S8010-Y70R), dark (S6010-Y50R), medium (S4010-Y30R), light (S2005-Y40R) and very light (S1005-Y50R). When analysing each manuscript, all the colours encountered in that document were written down in an Excel sheet. Then the number of occurrences of that particular colour is summed up for each period. Following this, the frequency of how often a particular colour is found in a specific period, percentage-wise, is calculated. Additional observations were also made, such as hue changes of the manuscript ink throughout the centuries.

3.2.2. Solubility tests

To determine the solvent suitable for possible conservation treatments, the inks were tested for solubility in 100 % deionised water, and 25 %, 50 %, 75 % and 100 % isopropyl alcohol. In each manuscript, we tested two examples of darkest brown inks and also the red lines used in accounting books for demarcating the columns and marginalia, if present.

3.2.3. Evaluation of ink corrosion

The level of ink corrosion was defined based on the IGI Condition Rating [22] examination under visible and UV light, according to the characteristics described in Table S1.

If there was no brown discolouration of the inked areas on the verso and no fluorescence, then the level was 0 or excellent; if there was observable discolouration, but still no fluorescence, then the level was 1 or good; if there was obvious darkening of the paper from ink on the verso and fluorescence starting to appear, then the level was 1,5 or satisfactory; if paper appears burnt, there is spreading of yellow discolouration around the ink lines and there is obvious fluorescence, the level is 2 or bad. The worst condition, 3 or loss of integrity, when there are cracks and loss of text, was not identified in this archive.

3.2.4. Analysis of the ink with test papers

Each object was tested twice in the darkest ink area with bathophenanthroline indicator paper strips (Indicator paper, GMW - Germany) to determine the presence of Fe^{2+} ions, following the procedures described in [34–36]. If the test paper turns pink, it means that the reaction was positive. The strips that showed no signs of Fe^{2+} ions were not retested with ascorbic acid to see if Fe^{3+} (ferric) ions were present.

Since copper is a stronger catalyst of cellulose oxidation than iron, copper test strips for the conservation community have been proposed ([17], pp.164–166), but this research has never resulted in a commercially available product. Therefore, to identify the presence of copper ions, a test with general copper test strips was tried – Cuprotesmo paper – produced by Macherey-Nagel [23]. A strip of test paper is cut, wetted slightly (it has to be damp but not too wet), and put over the inked line for 30 s. If the reaction is positive, a pink colour appears on the test paper. The intense pink indicates a strong reaction, while pale pink suggests that copper ions are present but not in large amounts.

4. Results and discussion

4.1. Paper support

4.1.1. Overall examination

4.1.1.1. Principal damages and their extent. The most prominent feature of the collection is that it is heavily soiled (Table S2). About half of it (51 %) would require surface cleaning. About 7 % of the whole collection shows obvious signs of microbial damage. Microbial assessment of this collection required specific analyses, which have been published elsewhere [37]. Gelatine-sized rag papers after 1800 and copybooks are the most affected by fungal attack. Dry cleaning and disinfection should take most of the conservation time to make the collection accessible.

A third of the collection (32 %) has some tears but only some would require mending for safe handling. Fortunately, only one object (less than 1 % of the collection) had signs of fire damage. Pressure-sensitive tape was found mostly in earlier volumes (2 % of the total), and metal fasteners will need to be removed only in 3 % of the objects.

Factors such as foxing, yellowing, and tide lines are not relevant for digitisation projects or general access but are indicators of issues related to paper stability. Foxing was found in half of the collection (47 %). Earlier papers, prior to 1800, are most heavily affected by foxing (64 %) and yellowing (29 % compared to 11 % of the second period 1800–1870 and 7 % of later period after 1870). Regarding water damage and tide lines, rag papers after 1800 are most affected (29 % compared to 14 % of the earlier period and 20 % of the later period after 1870). This leads to loss of sizing, which might cause general weakening of the paper support and binding as well as potentially promoting microbial attack. Rag papers after 1800 also show the highest percentage of fungal damage.

4.1.1.2. Paper thickness. Generally, papers of the same period are very much alike. Most papers within bindings are of medium thickness (0.10–0.20±0.05 mm thick). There are no thick writing papers in the collection. Copy papers are extremely thin, all of them are less than 0.10±0.05 mm. These copy papers are made from non-wood plant fibres and are intentionally unsized to transfer ink from one paper to another while wet.

Copy books, composed exclusively of copy papers, constitute a significant 29 % of the collection, while the rest 71 % are stationery bindings with accounting and legal content. Despite being so thin and slack-sized, copy papers in this collection are in good condition; there are few tears and little loss of media. There are some planar deformations caused by the application of humidity and pressure. The media do not seem to have affected the stability of this paper.

4.1.1.3. Water absorbency. Besides the copy papers, most of the other types of paper are hard-sized (Table S3). It was also observed that sizing can be jeopardized in case of water or mould damage; when the paper was tested in the same manuscript in clean areas and within tide lines or fungal deterioration, it was observed that in most cases there is greater water absorption in affected areas, which is often explained as a result of loss or degradation of sizing ([38], p. 75).

4.1.2. Surface pH

All the analysed papers are acidic, except some of the copy papers from the third quarter of the 19th century (Fig. 1). Gelatine-sized rag papers show pH values between pH 4.1–6.7 ± 0.1, and a larger proportion of objects have pH <5.5. It is known that aged paper is usually slightly acidic, but when pH falls below 5.5 the paper loses its permanence [39]. Rosin-sized papers have very low pH (from 3.3 ± 0.2 to 5.5 ± 0.2). It should be taken into consideration that surface pH values are usually lower than cold extraction pH values [16]. For rag papers, this difference could be larger than rosin-sized papers, since gelatine-alum sizing is usually on the surface as the paper is dipped into the size after it is made, while rosin papers are vat-sized, meaning size is mixed with the paper pulp prior to sheet production.

4.1.3. SurveNIR – pH

The pH values of papers determined using the SurveNIR instrument exhibit a trend consistent with measurements of surface pH values (Fig. 2). However, the correlation between the surface pH values of the papers and SurveNIR pH values is weak, primarily attributable to the high errors of determination associated with SurveNIR measurements.

A comparison of the pH results obtained by SurveNIR of the NST archive (Fig. 2) and those of the Classense library in Ravenna [11] shows that Italian gelatine-sized papers have much higher pH, mostly between 6.0 and 8.0, while the Bergen material, composed primarily of Dutch paper, is more acidic with pH values under 6.5. Paper made from the rosin-sized bleached chemical pulp in both collections is in the same pH range – between 3.5 and 6.5.

The overall low pH might be explained by the high concentration of alum, which hardens the size. Barrett [38] pointed out that the eight-fold increase of alum in 1700s' papers compared to 1600s' usually demonstrates high alum content – between 600 and 1600 ppm, compared to 200 ppm in papers from around 1600. It was also argued that possibly in the case of a high gelatine concentration (above 5.5 %) the effect of alum could be partially mitigated and result in more stable papers.

Although both libraries exhibit a general decrease in pH values after 1800, attributed to the introduction of rosin sizing in paper production, the decline in Bergen is not as pronounced. This is because older papers, dating back to before 1800, and even as far back as 1600, have pH values of 5.0.

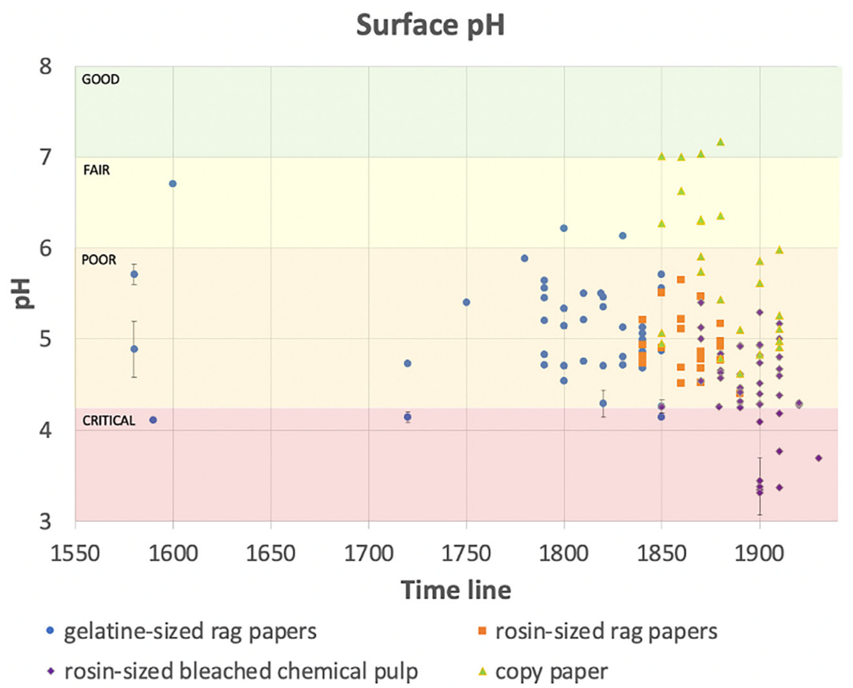

Fig. 1. Results of surface pH measurements: Surface pH for all 133 surveyed items of the Norwegian Sea Trade archive. Error bars indicate standard deviation. The pH categories are based on the SurveNIR manual [43].

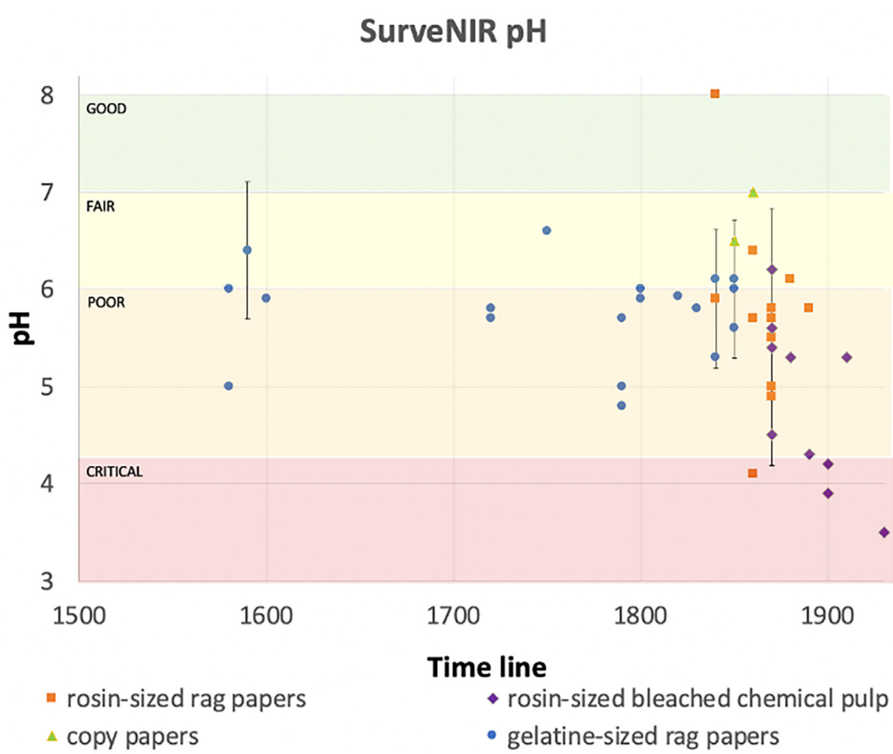

Fig. 2. pH of paper, determined by SurveNIR. Error bars indicate the instrumental errors of SurveNIR for this specific analysis [11]. The pH categories are based on the SurveNIR manual [43].

4.1.4. Paper composition types

Using SurveNIR, it was possible to identify four different types of paper in this collection (Fig. 3): gelatine-sized rag papers (1577–1850), rosin-sized rag papers (1840–1890), rosin-sized bleached chemical pulp papers (1872–1925) and unsized thin copy papers made of non-wood plant fibres (1851–1914). It should be pointed out, however, that SurveNIR does not distinguish between bleached and unbleached chemical pulp. Rosin-sized chemical pulp papers constitute the largest portion of the collection. Surprisingly, no groundwood papers were found (Fig. 4). According to SurveNIR categories, groundwood paper is defined as having more than 70 mg/g or 7 % lignin. However, according to Strlič et al., groundwood paper is defined with >50 mg/g of lignin [39]. This collection contains some books that fall into this intermediate cate-

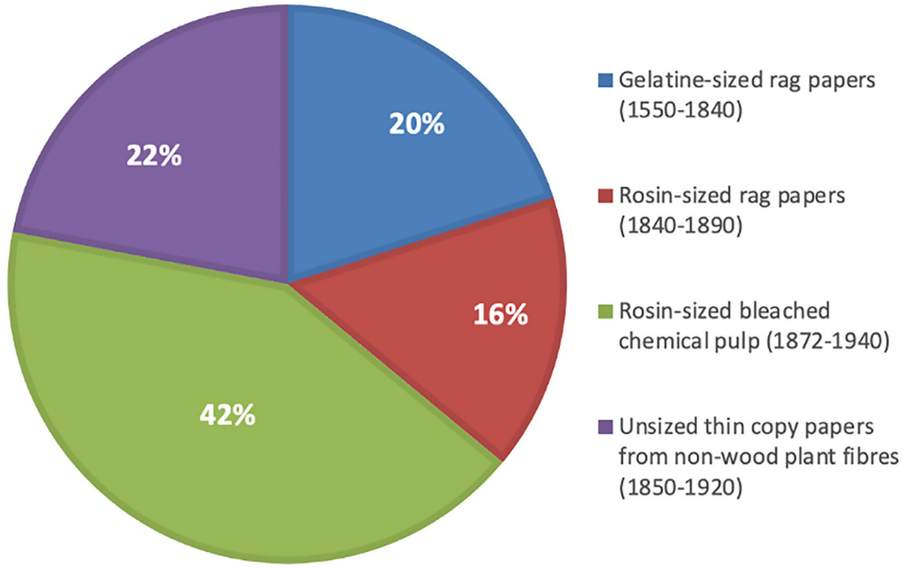

Fig. 3. Types of paper support found in the Norwegian Sea Trade archive, University of Bergen Library. Information based on the SurveNIR data.

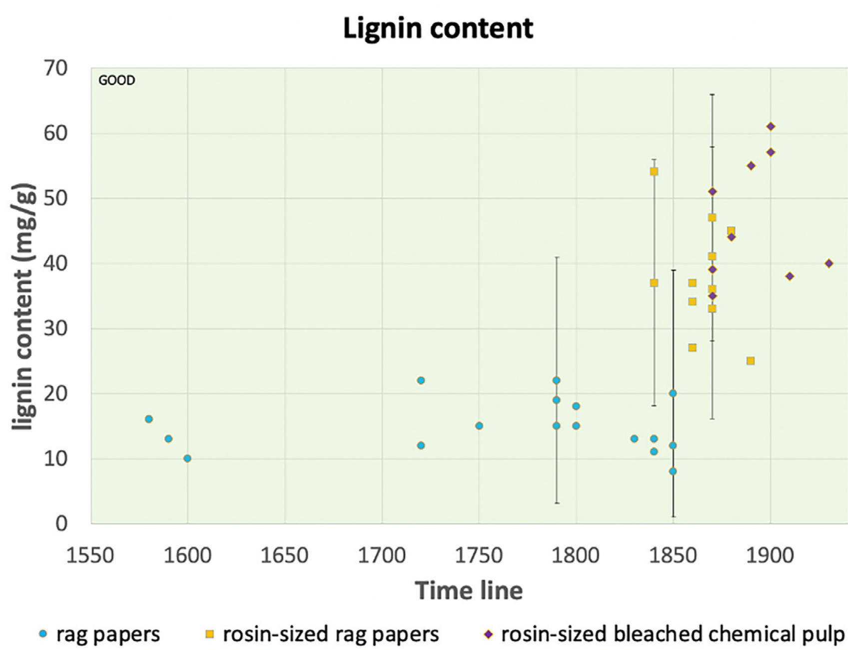

Fig. 4. Lignin content determined by SurveNIR. Error bars indicate the instrumental errors of SurveNIR for this specific analysis [11]. The category "Good" is based on the SurveNIR manual [43].

gory, of more than 5 % but less than 7 % of lignin content. They constitute 12 % of all tested items. These are notebooks and so-called "gesell" or apprentice books, that were used by journeymen to record the amount of fish each fisherman delivered; their paper has the highest lignin content between 51 and 61 mg/g. These books had the shortest working life span, as information from them would be entered into the next level of accounting, called Nordfarkladde. Possibly for that reason, cost could be spared for this type of manuscript.

Our spot testing on a subsample of papers until the early 19th century identified rag pulp (mainly flax-type fibres) (Figure S1). Some showed a slight reaction to phloroglucinol, turning light orange, which might indicate a small percentage of lignin. Flax fibres naturally contain lignin and SurveNIR measurements showed that rag papers have around 8–23 mg/g lignin content (Fig. 4). From around 1850, rag papers started to include cotton. Phloroglucinol could not detect lignin in these later rag papers, but SurveNIR showed similar amounts of lignin, within 20 mg/g, which can be related to the instrumental error associated with this measurement [11]. Papers after 1870 revealed the presence of rag pulp, but the presence of wood fibres was found inconclusive. Repeated testing, with a larger sample, will be required for the identification of bleached chemical pulp and confirmation of the SurveNIR results in this subsection.

4.1.5. SurveNIR – gelatine sizing

To compare the data collected in Bergen with other similar historical collections, a survey of the Classense library, Ravenna was used [11], since it also employed SurveNIR analysis. Protein content was found to be generally low in gelatine-sized rag papers (1.5–

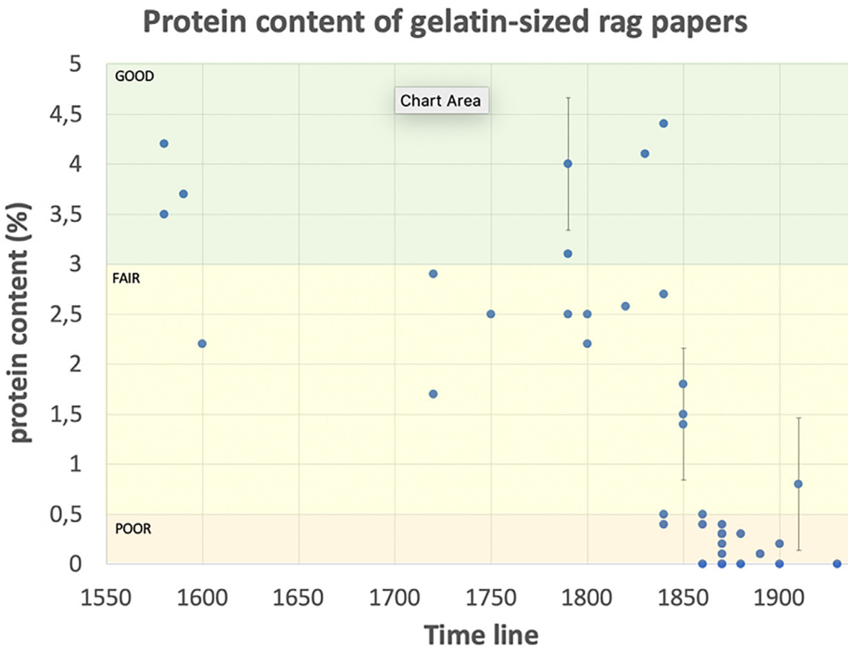

Fig. 5. Protein content determined by SurveNIR. Error bars indicate the instrumental errors of SurveNIR for this specific analysis [11]. The categories are based on the SurveNIR manual [43].

4.4 %) according to SurveNIR (Fig. 5). It can be seen that Italian papers of the same period usually have higher protein concentration, the majority between 3 and 6 %. The research on historical papers by Timothy Barett also pointed out the same trend, of generally much lower concentration of gelatine in German papers compared to Italian ones, and a strong decline of gelatine content to 1–2 % in Northern papers after 1600 [41]. In both libraries, Ravenna and Bergen, one could notice the same trend of declining protein content through the centuries.

According to SurveNIR categories, protein concentration over 3 % is considered good, while the Barrett research [41] showed that papers of the highest quality, 4 (good) and 5 (best) had an overage of 5 % protein concentration. Also, there has been observed in previous studies, a correlation between higher gelatine content (5.5 %) and higher pH and overall stability [42].

The very low protein concentration in papers after 1850 is due to the fact that these papers are mostly rosin-sized but the NIR still detects the presence of protein, albeit in very low concentration.

4.1.6. SurveNIR – rosin sizing

Rosin sizing appears in papers of this collection from 1840, first as sizing for rag pulp and then after 1872 for bleached chemical pulp. These papers show a high rosin content, reaching up to 7 mg/g; the highest concentration of rosin was found in papers between 1860 and 1880 (Fig. 6). In the Ravenna library, most rag and bleached chemical pulp have rosin content below 5 mg/g. Only groundwood papers show higher rosin concentration. Matija Strlič raises concern regarding rosin, not only because of its inherent acidity but because it tends to degrade via autooxidation in air and light and continues to acidify with time [40].

4.1.7. Mechanical stability

The mechanical stability of paper was measured by SurveNIR through tensile strength (TS), tensile strength folded (TFS) and degree of polymerisation (DP). TFS represents the tensile strength of the paper along the grain direction after having been uniformly folded mechanically [43]. TFS is comparable with the folding endurance test, which is considered one of the most sensitive of mechanical tests, as Havlínová et al. pointed out: “The difference in folding endurance is observable long before the changes in tensile strength” [44]. It can be argued that TS best represents the strength of the crystalline cellulose, while TFS might also include hemicelluloses and speak of the paper’s pliability. TFS was chosen for discussion as it could indicate the mechanical stability of paper better than TS.

4.1.7.1. Mechanical stability tensile strength folded (TFS). Except for copy papers, all the others are stationery papers, similar in thickness and sizing and thus are comparable. The analysed rag papers show an overall good TFS (Fig. 7), even though these values are lower (15–53 N) than the Italian rag papers from Ravenna Library (25–80 N) [11]. A characteristic feature of this collection distinct from the Ravenna library is that older papers, from the 16th to the middle of the 18th centuries are weaker, i.e. have lower TFS, while papers from the end of the 18th and early 19th century exhibit the most strength. Rosin-sized papers can fall very low, in the range of 5–32 N for Bergen and 7–50 N for Ravenna. This drop in TSF in the second half of the 19th century is characteristic of both collections.

4.1.7.2. Degree of polymerisation. The degree of polymerisation (DP) measures the average number of D-glucose monomer units within the cellulose polymer. The longer it is, the better the mechanical properties of the paper. Reference DP values in the SurveNIR database were obtained through viscometry [43]. Since viscometry cannot be performed for groundwood papers, this information on DP is not provided in the report of the Classense library. To make a comparison of the Bergen library with other collections, we used the data created by Strlič et al., in the analysis of European papers that lies at the base of the SurveNIR database [40, electronic supplementary material].

Comparing the two datasets (Fig. 8 and [40]), one can see similarities: DP falls within the same range for rag and chemical bleached pulp papers through the centuries. For rag papers, the average for the Bergen collection is 1678 and that for European paper research is around 1185; for chemical bleached pulp for Norwegian Sea Archive it is 1060 and for European paper research 715. There

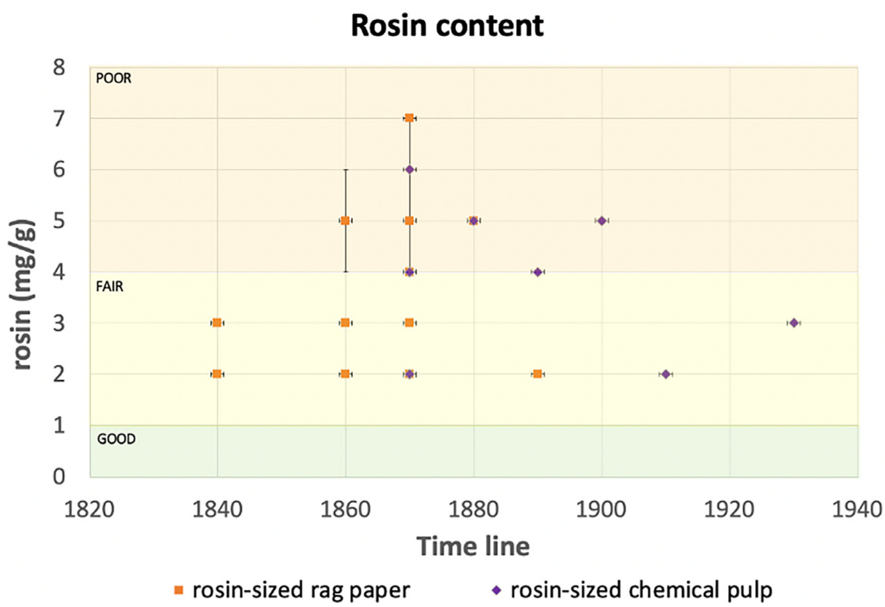

Fig. 6. Rosin sizing content determined by SurveNIR. Error bars indicate the instrumental errors of SurveNIR for this specific analysis [11]. The categories are based on the SurveNIR manual [43].

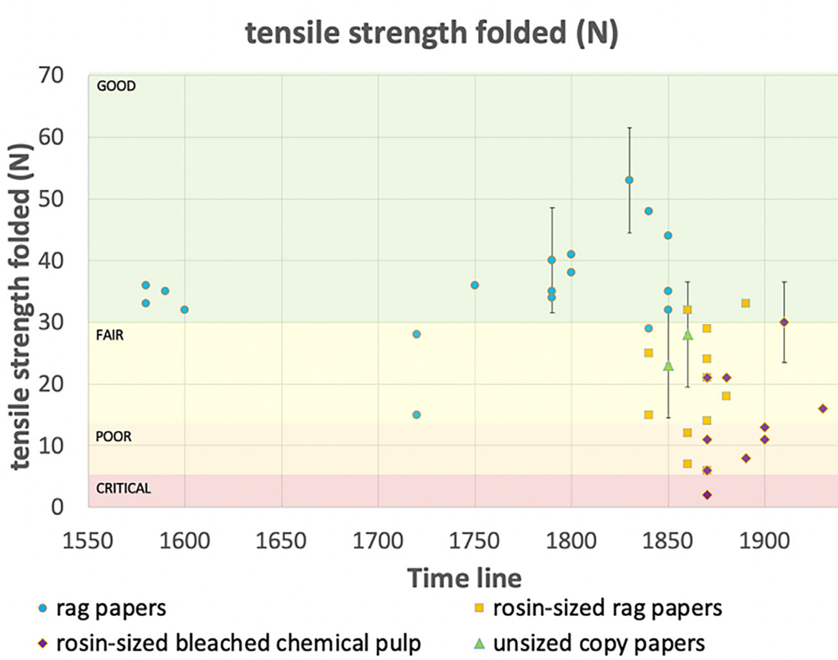

Fig. 7. Tensile strength folded (TFS), determined by SurveNIR. Error bars indicate the instrumental errors of SurveNIR for this specific analysis [11]. The categories are based on the SurveNIR manual [43].

are similar declining tendencies of the DP towards the late 19th century.

In theory, TSF and DP should be directly correlated; with the drop of DP, TSF should also decline. This tendency was also observed (R^2=0.5), but the best linear correlation between DP and TSF was found in rosin-sized rag papers (R^2=0.85). Perhaps other factors, such as variability of paper production, sizing, fillers, age, and effect of media among others, make this correlation less predictable for other paper types.

Typically, only a minority of papers exhibit critical or poor DP values. The majority of the papers tested demonstrate fair or good DP, indicating they maintain favourable mechanical qualities, as further supported by measurements of TSF, and are therefore safe to handle.

4.2. Manuscript inks

4.2.1. Visual examination

Accounting books are characterized by a very calligraphic and regular writing style. That is why very thick lines, over 1.5 mm thick (flourishes are not included here), in actual text are mostly not encountered (Table 1). Earlier ink is characterized by medium-thick lines (0.5–1.0 mm). By the 19th century, the amount of thin lines (<0.5 mm) doubled, which is explained by the invention of

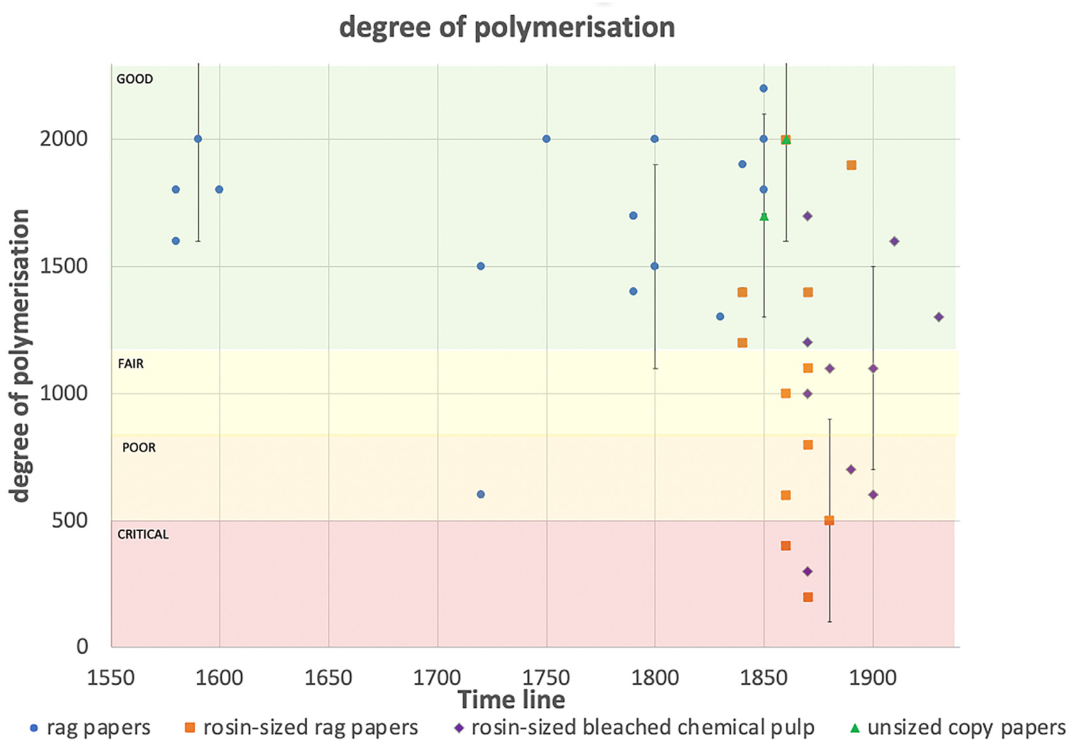

Fig. 8. Degree of polymerisation determined by SurveNIR. Error bars indicate the instrumental errors of SurveNIR for this specific analysis [11]. The categories are based on the SurveNIR manual [43].

Table 1
Line thickness found in each manuscript.

Line thickness	1500–1799 $n = 14$	1800 – 1869 $n = 45$	1870–1930 $N = 74$
very thick >1.5 mm	0 %	0 %	1 %
thick (1.0–1.5 mm)	7 %	13 %	12 %
medium (0.5–1.0 mm)	71 %	41 %	41 %
thin (<0.5 mm)	21 %	47 %	45 %

dip pen nibs, which, unlike goose feather quills, allowed the deposition of very regular thin lines.

In the earlier period, due to decorative calligraphic tendencies, there were elaborate titles and curlicues, and that is why extra thick lines, are found there the most (Table S5). There was a steady decline of those flourishes by the first half of the 19th century and by the end of the century, they completely disappeared. Thus, there is a very specific progression observed from thickly applied ink in the earlier period, before 1800, to a much thinner application of ink in the late 19th century.

In earlier manuscripts, due to the abundant use of thick lines in baroque flourishes and titles, the ink tends to be thickly applied, and has corroded to a greater extent, and that is why the percentage of very dark ink for the earlier period is larger than in the first half of the 19th century by 10 % (Table S6). The colour of the ink shifts from dark brown to cold deep black by the end of the 19th century (Fig. 3). At that time, the predominance of dark ink was mostly due to added dyes, like aniline-based synthetic dyes, rather than the degradation of the ink.

To define treatment options, which could include aqueous and organic solvents, the solubility of brown/black and also red inks (these used in demarcating column lines and marginalia) were tested for water and alcohol (isopropanol).

It was found that in the case of earlier inks, scribes often wrote with partially oxidized ink, which left particles of ink on the surface of the paper. Testing with a blotter moistened with water or 25 % alcohol often resulted in some transfer of particles of ink to the blotter (Table S7). In some rare cases, there was only ink as solid particles that could be so easily lifted that almost nothing could be seen underneath. Until 1870, red demarcating lines of the manuscripts seem to have been drawn by scribes themselves. Although the colouring agent has not yet been identified, this red ink seems stable and mostly not soluble in water or alcohol.

That suggests that treatment of those earlier manuscripts would ideally employ solutions of 50 % alcohol or more on the suction table or possibly on the blotter, to avoid immersion or other types of washing where water on the surface of the manuscript might accidentally remove those loosely adhered particles.

After 1870, ink solubility changes. Liquid ink is easily transferred onto the blotter either with water or alcohol, including sometimes up to 75 % alcohol. This can be explained by the addition of dyes, especially synthetic aniline dyes, which are soluble in alcohol. By that period accounting books were being printed and bound by commercial publishing companies. Printed red lines also show greater solubility in both water and alcohol. In cases of water-damaged pages, the spreading of those red lines can be easily observed. This complicates the treatment options. In cases where ink corrosion is prominent, only local treatment of specific ligatures with 100 % alcohol solution on the suction table could be advised.

4.2.2. Presence of iron and copper in the inks

Testing for the presence of Fe^{2+} ions (Table S8) showed that in some manuscripts Fe^{2+} ions were not detected in the ink. In one case, in a manuscript written after 1753, a negative reaction of Fe^{2+} ion gave a positive reaction to copper. Distribution of the amount of ferrous ions is more or less even throughout the manuscripts and periods. The only exception is found in copy books, 69 % of them having no or very little ferrous ions detected.

Table 2
Overall ink condition throughout the collection.

Category	Percent
0 – best	22%
1 – good	47%
1.5 – exhibit signs of corrosion	14%
2 – corroded	16%
3 – lack of integrity	0%

These tests are usually used to prove the presence of IGI. However, positive tests for Fe^{2+}ions can be also obtained with other brown inks besides iron-gall inks, such as bistre or logwood, because they can contain small amounts of iron ions due to storage in iron containers or iron writing or drawing tools ([17], p.153). Therefore, to accurately determine the presence of IGI, additional analyses have to be performed, which are beyond the scope of this article and will be covered in our future research.

Testing of inks for the presence of copper ions is important as it has been proved that the overall mass fraction of copper content in IGI manuscripts sometimes reaches that of iron, while in comparison to iron, it is even much more catalytically active in inducing oxidative degradation of cellulose [24].

Cuporetesmo paper tests for the detection of both Cu^{2+} and Cu^{+} ions were used as there are no other test papers developed for paper conservation purposes available on the market at the moment. However, the test was designed for other purposes and therefore has to be evaluated thoroughly before it could be considered reliable. Only three manuscripts (1753–1829) tested positive; in the rest of the documents, copper was not detected with the method used.

4.3.3. Overall condition of manuscript inks

Summarising all the results above, and according to the method described in 3.2.3, the condition of the manuscript ink was assessed (Table 2). Overall, the writing inks in the analysed collection are not strongly corroded and may be rated on average condition 1 or good, but 20 % of the collection does show signs of iron corrosion (condition levels 1.5 and 2). However, there are none of level 3, meaning there are no cracks or missing elements. Most ink corrosion (levels 1.5 and 2) is found in the category of gelatine-sized rag papers, and those documents from before 1800 appear to be more corroded (level 2 – 29 %) than those after 1800 (Level 2 – 19 %) (Table S9). Also, thin copy papers have a high percentage of levels 1.5 and 2 due to the thinness of the paper (level 2 −20 %).

Except for copy papers, all other papers are of medium thickness (0.1–0.2 mm) and most are hard-sized. It might be suggested that the predominance of hard-sized and relatively thick papers is one of the contributing factors to the good condition of paper support, not significantly affected by corrosive media.

Copy papers are mostly not affected by ink corrosion. Written ink does not show signs of fading, while the transferred ink of copy papers does have issues of legibility. This could be either the result of insufficient ink used in the original for copying purposes or of fading.

The good overall condition of ink in the analysed collection may be due to the absence of copper (or at least low amounts, undetected by the used method), and possibly cool storage conditions, since the lower the temperature the slower the rate of the chemical reactions [47]. Also, temperatures in Bergen do not change significantly throughout the year, varying between ca. 5 °C and 18 °C degrees, with an average temperature of 8 °C. In addition, during the last 80 years, the collection has been stored in a dry environment, and in summer the ambient temperature does not exceed 19–20 °C.

5. Conclusions

The final goal of this survey was to provide recommendations for storage and to establish measures to make the NST archive accessible. This investigation showed that the overall condition is good, as the papers and manuscript inks maintain their integrity. However, for the collection to be fully accessible, a certain amount of conservation work will be required.

Based on pH values, mechanical properties, gelatine, and rosin and lignin content, the overall condition for all the papers is considered “good”, as there are certain damages but the material can be normally handled without risk of further damage. Most rag papers are of poor to fair quality, based on the system proposed by Timothy Barrett [41]: pH $\leq$ 6.0 and low protein content ($<$ 5 %). Alum-rosin-sized papers in this collection are similar to the ones in other collections, like the Classense library, although the pH is slightly lower. No groundwood paper, as defined by SurveNIR, was found in this collection.

The mechanical integrity of the papers is satisfactory; the DP for gelatine-sized rag papers is good while it is rather low for rosin-sized papers. TSF is in the lower range for all papers compared to papers of a similar period in the Classense library, and as shown by the SurveNIR project on the development of a database on historic European paper properties [40].

The documents containing Fe^{2+} are not severely corroded, although 20 % of the collection does show signs of corrosion (levels 1.5 and 2) that might require local treatment in the future. The majority of books requiring local anti-oxidative treatment are gelatine-sized rag papers from before 1800 and some after 1800. Most copy papers (69 %) did not show ferrous ions, which might suggest another type of media besides IGI.

Older papers, from before 1800, are generally weaker compared to the papers of the same period in the Classense library. This can be seen in lower TSF. This can be additionally observed in such signs of degradation as foxing and yellowing, and may be explained by lower paper quality, acidity, ageing, and higher degree of ink corrosion.

The survey also showed that for both preventive and remedial conservation it is important to consider the period of production of the manuscript as materials and techniques of paper supports and inks change. Three main groups can be traced:

- Before 1800 one finds mostly handmade gelatine-sized rag paper, and ink lines were thickly applied;
- After 1840, when rosin was introduced into the sizing of rag paper, leading to further lowering of paper pH;
- After 1870 when alum-rosin bleached chemical pulp became predominant, dyes were being added to the manuscript inks and marginalia, making media more sensitive to alcohol.

Most of the conservation treatment should consist of dry cleaning, as half of the collection is severely soiled. Treatment would also consist of disinfection, as 7 % of the collection shows signs of fungal attack.

The solubility of calligraphic ink, demarcating lines and marginalia showed that any wet or solvent treatments should be at least 50 % alcohol solution for manuscripts before 1870, and in 100 % alcohol solution for a later period. The treatment should be performed either on the blotter, rigid hydro-gel or preferably on the suction table to prevent the migration of loose ink particles.

At present, the archive is stored at 16–18 °C, 40–50 % RH. Research has shown that with each 5-degree drop the lifetime of a paper object doubles [45], thus for long-term storage of library material, cooler temperatures, c. 10 °C, is preferred [46]. During her presentation at the IADA conference, Marie Vest from the Royal Danish Library showed that storage of acidic paper requires a lower temperature, around 12 °C [47]. Folger Libraryś permanent storage is set for example at 13–14 °C, 40–55 % RH [48]. Thus, considering the high value of the collection and high acidity of the paper, the archive should be ideally kept in cool storage (between 10 and 14 °C) and low and stable relative humidity, between 40 and 55 %RH. Since all of the documents, including rag paper were found to be acidic, i.e. below pH 6.0, it is proposed to store all of the collection without exception in a cool environment.

Because of the observed acidity, methods of non-aqueous in situ deacidification should be considered. A few selected items, mostly gelatine-sized rag and copy papers will need non-aqueous in situ antioxidant treatment on the suction disc.

Regarding digitisation, the project should not be difficult as the format is consistent, there are minimal or no inserts, and little use of metal fasteners and pressure-sensitive tape ([7], p. 65). Bindings are mostly stable and very similar. Only a few earlier historical and most valuable items would require special handling.

Copybooks should be digitalised last as they would require more preparation work: the text is not fully legible and further research in multispectral imaging would be necessary to improve the contrast of the lighter lines.

Further research will be carried out to investigate the composition of manuscript ink via XRF and Raman spectroscopy.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.culher.2024.05.001.

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