Convergence and Divergence of Some Methods Used to Assess the State of Preservation of Paper in Old Books

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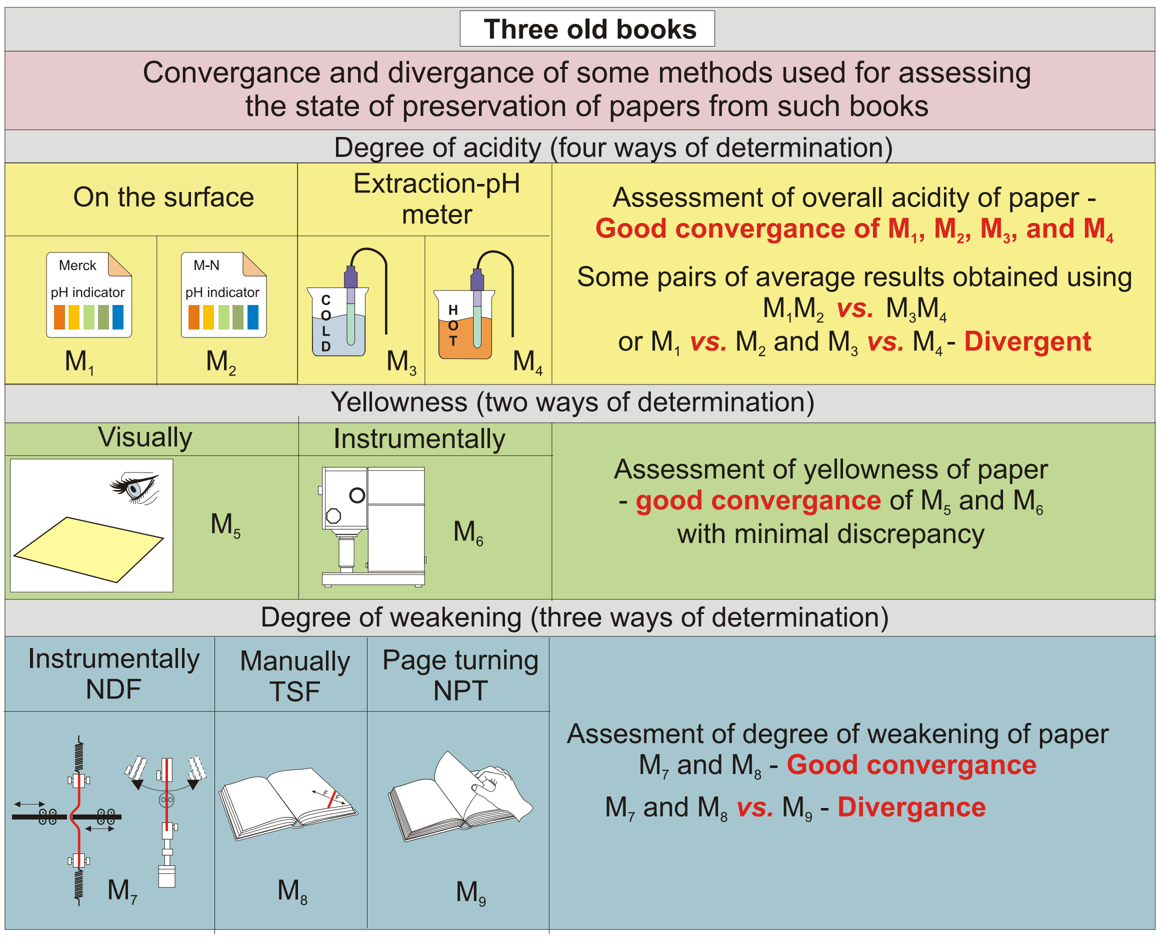
GRAPHICAL ABSTRACT



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The convergence or divergence of results for pH, yellowness, and mechanical strength were considered for papers from three old books. Good convergence was found for pH measurement results from the three books. The lack of convergence in some pH measurements between the surface method and the extraction–pH meter method may result from differences in pH of individual pages of old books, inherently lower pH values obtained using the surface methods, as well as from lower pH values measured in extracts of old papers prepared under hot conditions using a pH meter. Yellowness testing revealed overall good level of convergence between visual assessment and instrumental measurement (Elrepho 2000 apparatus). Strength tests indicated convergence between the results of the number of double folds (NDF) and tensile–after–six folds (TSF) tests, while divergence was observed when these methods were compared with the number of page turns (NPT) test. High NPT values may result from the papers of the examined books retaining yet relatively significant resistance to tensile and tearing stresses. Further research is needed to explain the relatively small damage to book pages in the NPT test in contrast to alarming results of the NDF and TSF tests.

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Keywords: Old books; Paper; pH; Yellowness; Mechanical strength; Convergence; Divergence

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INTRODUCTION

The assessment of the condition of library and archival collections is an important issue from the perspective of their protection against degradation. This particularly concerns paper-based collections from the late 19th century and the first half of the 20th century. Such degradation usually begins at the edges of pages and progresses inward into books, folders, and newspapers, leading to paper darkening and a loss of mechanical strength, ultimately resulting in its disintegration (Zeisler *et al*. 1993; Dąbrowski 1999, 2004a, 2005; Durovič *et al*. 2001; Area and Cheradame 2011; Hubbe *et al*. 2023).

The process of paper degradation is a complex issue. It results from the influence of numerous destructive factors, which can be divided into two main groups: internal factors and external factors (Jabłońska and Strzelczyk 2007). Internal factors include the technological conditions of its production such as type of pulp, sizing agent, filler, and other bulk additives used in papermaking, the presence of metal ions as well as the method of its surface refining (Jakucewicz 1990; Zou *et al.* 1993; Letnar and Vodopives 1997; Area and Cheradame 2011; Hubbe *et al*. 2023; Durovic 2001; Barański *et al.* 2003; Area and Cheradame 2011; Jablonsky *et al*. 2020; Hubbe *et al*. 2023).

External factors include the activity of microorganisms (Jamer 1948; Jabłońska and Strzelczyk 2007; Jacob *et al.* 2017), improper air parameters (lack of free air circulation, excessive humidity, and contamination) (Jabłońska and Strzelczyk 2007; Area and Cheradame 2011; Hubbe *et al*. 2023), the influence of sunlight, artificial light, and temperature, chemical air pollution (Garnagul and Zou 1994; Havermans 1995; Begin *et al.* 1999; Jabłońska and Strzelczyk 2007; Area and Cheradame 2011; Hubbe *et al*. 2023), migration of printing ink components and substances from paper binding, and natural disasters (Jabłońska and Strzelczyk 2007).

The primary cause of the degradation of papers produced during the industrial era is considered to be the use of aluminum sulfate as a coagulant for rosin sizing. As a salt of a strong acid and a weak base, it undergoes hydrolysis within the paper, leading to its acidity (Garlick 1986; Barański *et al.* 2003; Dąbrowski and Gonera 2005; Chamberlain 2007; Area and Cheradame 2011; Kołątaj 2020; Hubbe *et al*. 2023).

This method of paper sizing began to be used at the beginning of the 19th century and continued to be applied extensively throughout the 20th century. However, papers sized in an acidic environment proved to be non-durable, which led to increased interest in the paper industry in producing papers under alkaline conditions—first in Europe in the early 1950s, and later in the United States in the late 1970s. This shift was associated with the use of precipitated calcium carbonate as a filler, which could be cheaper than kaolin from Cornwall (Europe) and the state of New Georgia (USA), as well as with the availability of reactive synthetic sizing agents such as AKD and ASA (Dąbrowski and Gonera 2005). Papers produced using this technology include those that are referred to as permanent document and archival papers and should meet the requirements of the many standards devoted to this issue *e.g.*, PN-EN ISO 9706 and PN-ISO 10716:2009P standards, ensuring the possibility of archiving printed materials age-resistant papers for over 200 years (Wandelt 1999; Dąbrowski and Gonera 2005).

Concerns about the durability of papers produced using aluminum sulfate as a coagulant for rosin sizing were expressed relatively early, already at the end of the 19th century (Jurkiewicz and Weinberg 1887). At that time, however, it was the only technology suitable for mass paper production due to its low cost and required efficiency (Wandelt 1999). Only the invention of alkaline sizing made it possible to produce papers resistant to yellowing and loss of mechanical strength (Paiste 1981).

Large-scale efforts have also been undertaken to halt the degradation of books, newspapers, and documents in which papers were sized in an acidic environment and produced with the use of groundwood, as well as to improve their condition (Potrzebnicka 2019). These measures include assessing the state of preservation of different types of paper publications in library, archival, and museum collections, as well as deacidification, disinfection (fungal removal), strengthening, and improvement of storage conditions (Sobucki *et al*. 2003; Rams *et al*. 2004; Dąbrowski 2004b; Sobucki *et al*. 2008; Zervos and Alexopoulous 2015; Hubbe *et al.* 2017, 2018; Horst *et al*. 2020; Malesič *et al*. 2022).

Various methods are used to assess the condition of paper documents and book papers. These can be divided into non-destructive methods or those causing only minimal damage to paper objects (Hall *et al.* 2019), typically applied in conservation practice (*e.g.*, surface pH measurement, brittleness test, cantilever test, visual assessment of paper yellowing), and destructive methods, which require the use of fragments or even several sheets of paper and are applied in scientific research on paper conservation [*e.g.*, pH measurement using the extraction–pH meter method, number of double folds (folding endurance), tensile strength, tear resistance, tensile strength after folding] (Zervos and Moropoulou 2006).

This article addresses the problem of convergence or divergence of three properties of papers from books B_1 (the book form 1928), B_2 (1892), and B_3 (1933), namely:

• pH determined using the surface method with litmus papers from two manufacturers, as well as the extraction–pH meter method employing both cold and hot water extraction of paper samples,

• the degree of paper yellowness assessed visually and using an Elrepho 2000 spectrophotometer,

• and their mechanical strength, evaluated by means of the number of double folds (NDF) test, the tensile strength after six folds (TSF) test (like in Standford method, Sobucki 2003), and the number of page turns (NPT) test.

EXPERIMENTAL

General Characteristics of the Books Selected for the Study

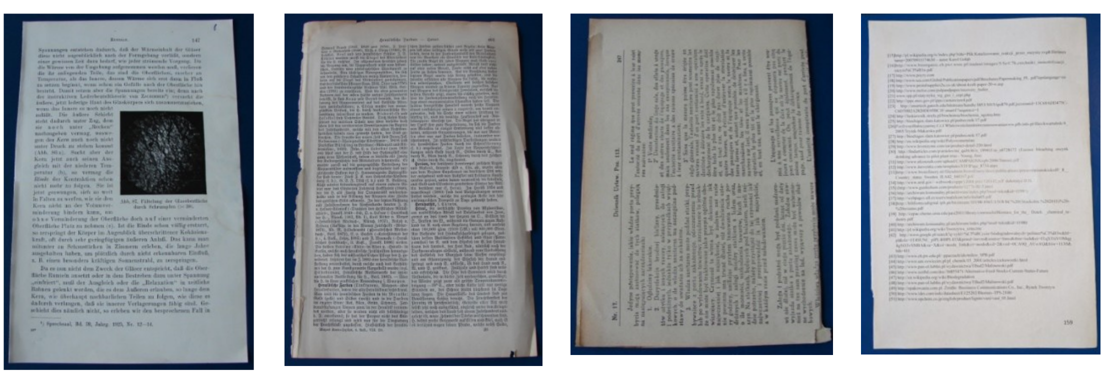
Since destructive testing of books (*e.g.*, tear strength, tear resistance, *etc.*) was planned, the study sought books that were worthless. The books selected for testing came from a library that had removed them from their inventory. Another reasoning behind the author's research was to select books that differ significantly in the year of publication. Given a choice of several books withdrawn from the library, old books B_1, B_2, and B_3 were chosen.

Book 1 (B_1): year of publication – 1928; place of publication – Germany; paper basis weight – 90 g/m²

Book 2 (B_2): year of publication – 1892; place of publication – Germany; paper basis weight – 70 g/m²

Book 3 (B_3): year of publication – 1933; place of publication – Poland; paper basis weight – 60 g/m²

To compare the properties of the paper of these books and modern book paper, the paper from this type of book marked in the text of the article as CBP (Contemporary Book Paper) was used. It came from a book published in 2012 in Poland. Its basis weight was 90 g/m² and was similar to photocopy paper.



B_1 B_2 B_3 CBP

Fig. 1. Pages of books B_1, B_2, B_3, and CPB

Measurement of Paper pH

Method 1

In this method, litmus papers were used to determine the pH of the paper in old books. In the literature of the subject, pH measurement using this approach is referred to as the *contact method*. In this study, the pH of the paper was measured in a manner similar to that described by Sobucki (2008). A page of the book was placed on a glass plate. Then, 0.1 mL of water was drawn into a 1 mL pipette, after which the water was applied with the pipette to the margin of the book page in an area not covered by print, over a field approximately 4 cm long and up to 1 cm wide. Next, a stopwatch was started for 120 seconds to allow the water to be absorbed into the paper structure and for its pH to equilibrate with that of the paper. After this time, a strip of litmus paper was placed on the moistened area of the paper surface and pressed against it for 30 seconds with the aid of a microscope glass slide. The pH value was then read from the color scale provided with the litmus paper set.

Two types of water were used to moisten the papers of books B_1, B_2, and B_3:

- Distilled water with a pH of 6.4 (W_1).
- Distilled water alkalized with $NaHCO_3$ to a pH of 7.0, labeled as W_2.

Litmus papers from Macherey-Nagel (M-N) (measurement range: 3.1 to 8.3 pH units; jump**:** 0.4 units) and Merck (MK) (measurement range: 2.5 to 7.0 pH units; jump: 0.5 units) were used as pH indicators. The pH of seven pages was measured with each type of litmus paper for books B_1, B_2, and B_3.

Method 2

In this method, the pH of papers from books B_1, B_2, and B_3 was determined using the extraction–pH meter method. This involved using filtrates obtained from the extraction of cut pieces of paper taken from the margins of these books, with pH measured by immersing the electrode of a pH meter in the extracts. Extracts were obtained using both cold (T = 23 °C) and hot (T = 100 °C) extraction of paper samples. The procedures for extraction and pH measurement followed ISO 6588-1:2021 and ISO 6588-2:2021 standards, except that the pH of distilled water W_1 was 6.4 instead of the recommended 6.8 to 7.3 units. As in Method 1, two types of distilled water (W_1 and W_2) were used in the study. Three cold and three hot extractions were performed using six paper samples from books B_1, B_2, and B_3 for each kind of water. Therefore, the results presented in Table 1 concerning the pH of the cold and hot extracts are averages of three extractions and three pH measurements.

Degree of Paper Yellowness

Method 1

This method was visual in nature. In this approach, the degree of yellowness (Y_V) was expressed based on the subjective assessment of the author of this publication using a scale where: 0 – no yellowing, 1 – low intensity of yellowness, 2 – medium intensity yellowness, and 3 – high intensity of yellowness. The assessment was carried out by placing several pages from books B_1, B_2, and B_3 on a laboratory table previously covered with black cardboard. The observation of the degree of yellowing of papers was conducted during the summer of 2024, between 2:00 and 3:00 PM, in a laboratory with windows facing east.

Method 2

This method involved determining the yellowness index according to ISO 11475:2017. Measurements were performed on the wider margins of several pages from books B_1, B_2, and B_3, stacked to achieve total opacity. Tests were conducted using an Elrepho 2000 instrument (Datacolor, Switzerland) without UV, measured under D65/10° conditions at a wavelength of 457 nm. In the results, this index is referred to as Y_E, *i.e.*, yellowness determined using the Elrepho 2000.

Degree of Paper Weakening

Method 1

This method involved determining the number of double folds (NDF). The test was carried out using a Schopper apparatus manufactured by J. Hengstler K.G. Zählerfabrik, Aldingen, Kr. Tuttlingen, and a Köhler–Molin apparatus produced by AB Lorentzen & Wettre, Stockholm, Sweden. The load applied to the paper strip in these devices was 500 g and 50 g, respectively. The characteristics of these apparatus types were described by Kahlson and Märtensson (1964). Tests were conducted in accordance with ISO 5626. The result represents the number of double folds a paper strip can withstand before breaking. This parameter was determined for paper strips from selected books in both the MD (machine direction) and CD (cross direction) taken from page margins. Prior to testing, the samples were conditioned for 24 hours in a room with relative humidity of 50% ± 2% and a temperature of 23°C.

Method 2

This test is referred to as "tensile-after-folding." The procedure was:

- Folding a corner of a page onto itself and pressing the crease between finger and thumb.
- Folding the corner back in the opposite direction along the same crease and pressing again.
- Repeating this two more times (six folds in total).
- Subjecting the paper to a tensile test along the fold.

In this study, this method is referred to as "tensile after six folds" (TSF). It is commonly used in conservation research for rapid assessment of the condition of paper in old books (Dąbrowski 2004a,b, 2007).

Method 3

This method involved turning three selected undamaged pages from books B_1, B_2, and B_3 1,000 times each (nine pages total). During turning, the condition of each page was examined. If damage was first noticed (e.g., tearing or loss of a fragment from the edge), the flip number was recorded, and flipping continued until 1,000 turns were completed. Photographs show the extent of damage after 1,000 turns. The rate of turning was approximately 35 turns per minute.

Determination of the Fiber Composition of Paper

A dry sample of paper was collected from each object. Prior to identification, samples were boiled in distilled water to remove contaminants and disintegrated. Analysis was based on fiber color in Herzberg's reagent and their morphological characteristics. Two comparative slides were prepared for each sample. A Biolar T transmitted light biological microscope was used.

Determination of Additional Paper Properties

- Tensile strength index of paper according to ISO 1924-2 using a Zwick/Roell apparatus.
- Tear resistance index of paper according to PN-EN ISO 1974 using a ProTear (Thwing-Albert) apparatus.

RESULTS AND DISCUSSION

pH of Paper

One of the important properties of paper from old books, for which the convergence and divergence of evaluation methods were analyzed, was its pH. Monitoring the degree of acidity is particularly important, as an acidic environment accelerates the degradation of cellulose, leading to fiber weakening, loss of flexibility, and embrittlement of the papers. Monitoring of papers pH levels allows for the early detection of risks of their weakening and the implementation of appropriate preventive or intervention measures aimed at extending of their durability (Długosz-Jasińska 2025).

Table 1. Results of pH Determinations of Papers from Books B_1, B_2, and B_3 Using Different Methods

Book, index / Method	B_1	$\bar{x}$ Δ_1 Δ_2	B_2	$\bar{x}$ Δ_1 Δ_2	B_3	$\bar{x}$ Δ_1 Δ_2
	W_1 – distilled water, pH 6.4					
MK (M_1)	5.0 ±0.28	5.5/ 1.55/ 1.0	4.0 ±0.20	4.2/ 0.7/ 0.4	4,0 ±0.10	4.15/ 0.2/ 0.3
M-N (M_2)	6.0 ±0.14		4.4±0.25		4.3 ±0.33	
Cold extraction–pH meter (M_3)	7.3 ±0.20	7.05/ 1.55/ 0.5	5.3 ±0.24	4.9/ 0.7/ 0.8	4.6 ±0.16	4.35/ 0.2/ 0.5
Hot-extraction–pH meter (M_4)	6.8 ±0.12		4.5 ±0.11		4.1 ±0.13	
	W_2 – distilled water alkalized with Na_2CO_3 to pH 7.0					
MK (M_1)	6.0 ±0.20	6.2/ 0.3 0.4	4.0 ±0,10	4.3/ 1.3/ 0.6	3.7 ±0.20	3.7/ 1.35/ 0.0
M-N (M_2)	6.4 ±0.20		4.6 ±0.31		3.7 ±0.25	
Cold extraction–pH meter (M_3)	6.9±0.21	6.5/ 0.3/ 0.8	6.2 ±0.11	5.6/ 1.3/ 1.2	5.4±0.15	5.05/ 1.35/ 0.7
Hot-extraction–pH meter (M_4)	6.1 ±0.26		5.0±0.18		4.7 ±0.14	

$\bar{x}$ – arithmetic mean of the pH measurement results using Merck and MN papers and such mean of the pH measurement performed using pH meter of cold and hot water extracts of papers of books B_1, B_2, and B_3
Δ_1 – the difference between the mean values $\bar{x}$ of pH determinations using the surface and extraction-pH meter methods. For example, for book B_1: 7.05-5.5= 1.55, *etc*.
Δ_2 – the difference in pH determined with Merck and MN papers and with a pH meter for aqueous extracts obtained in cold and hot conditions. For example, for a book B_1: 6.0-5.0 = 1.0; 7.3-6.8 = 0.5, *etc*.

This property can be assessed using non-destructive methods (*e.g.*, litmus paper, flat-tipped glass calomel electrode, and polyaniline electrode) (Čabalová *et al*. 2017) and destructive methods, such as measuring the pH of cold or hot (Barrow 1964) or hot (Bobu and Poppel 1988) aqueous extracts of paper samples. As described in the methodology, this study used two approaches to examine the agreement of pH measurements: surface pH determination using litmus papers produced by two manufacturers, and pH measurement of aqueous extracts obtained by cold and hot extraction of paper samples using a pH meter.

The results of pH determination for papers from books B_1, B_2, and B_3 obtained by these methods are presented in Table 1. Based on these results, the convergence or divergence of pH determination methods was evaluated in terms of:

- the overall similarity in the degree of acidity of these papers measured by different methods (comparison of acidity levels for papers from books B_1, B_2, and B_3),
- differences in the mean pH values obtained from two ways of its surface determination and two the extraction–pH meter ways of determination (comparison of the Δ_1 index),
- and differences in mean pH values measured using litmus papers from Merck and Macherey-Nagel, as well as by pH measurement of cold and hot extracts (comparison of the Δ_2 index).

The tests were carried out using two types of distilled water: non-alkalized distilled water (W_1) with a pH of 6.4, and distilled water alkalized with small amount of Na_2CO_3 to a pH of 7.0 (W_2).

The results of pH determination for papers from books B_1, B_2, and B_3 using the above-mentioned methods are presented in Table 1.

Determining the convergence of results in assessing the overall degree of acidity of papers from books B_1, B_2, and B_3 using different methods allows for a comparison of trends in the $\bar{x}$ index, *i.e.*, the mean pH values obtained by the surface method (litmus papers) and the extraction–pH meter method, shown in Fig. 2.

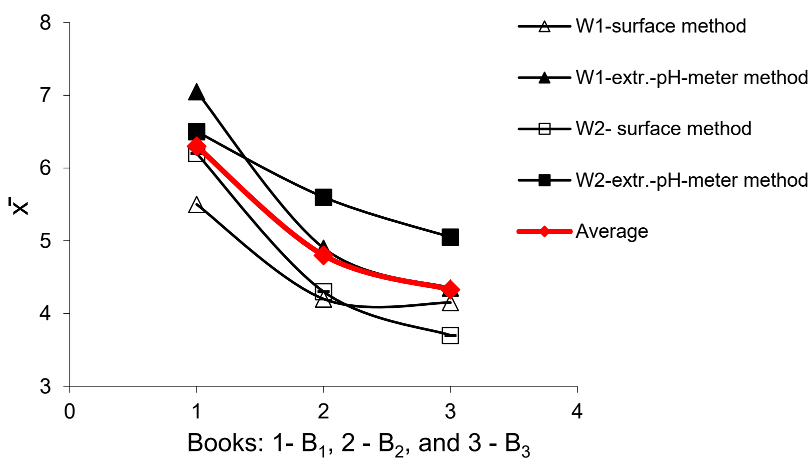


Fig. 2. Convergence and divergence of measurements of the overall pH of papers from Books B_1, B_2, and B_3

As shown in Figure 2, the results of pH measurements for books B_1, B_2, and B_3 using the surface and extraction–pH meter methods were convergent in terms of the overall pH. They indicate (red line representing the average values from four measurement methods) that book B_1 was the least acidic, book B_2 was more acidic, and book B_3 was the most acidic.

The data in Figure 2 also allow for an assessment of the convergence and divergence of pH measurement results for papers from the selected books obtained by the surface and extraction–pH meter methods. This type of analysis is possible by comparing the range of variation of the $\bar{x}$ index for each book. From Fig. 2, it can be seen that the mean values obtained by the surface method and the extraction–pH meter method for books B_1, B_2, and B_3 differed by approximately 1.55 units, 1.4 units, and 1.4 units, respectively. Differences of this magnitude in the measured pH values of paper could be considered large. However, Fig. 2 shows that most of the pH measurements for books B_1, B_2, and B_3 fell within ranges of approximately 6.5, 4.75, and 4.3 pH units, respectively, which in conservation practice are regarded as slight, strong, and very strong (catastrophic) acidification of paper (Winiarczyk and Rosa 2007; Rosa 2007).

The convergence and divergence of pH results obtained by the surface and extraction–pH meter methods can also be compared by compiling the differences in $\bar{x}$ values from these measurements, *i.e.*, the Δ_1 index. These differences are presented in Fig. 3.

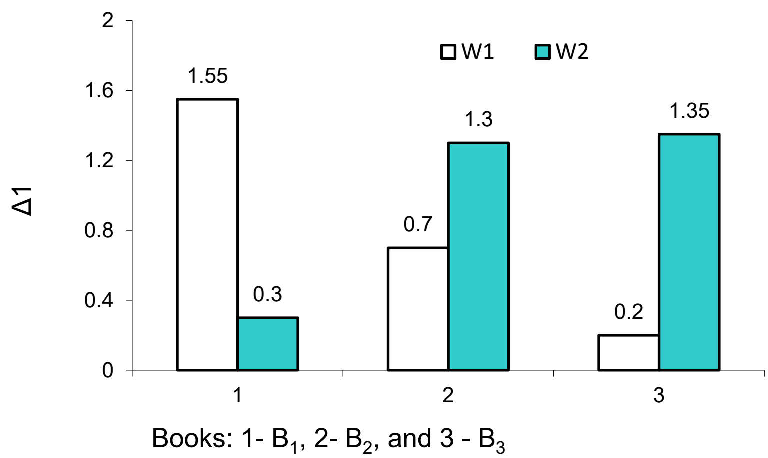


Fig. 3. Differences in $\bar{x}$ Values of Papers from books B_1, B_2, and B_3 measured using Merck and Macherey-Nagel litmus papers and a pH meter with cold and hot aqueous extracts

As shown in Fig. 3, for measurements conducted using water W_1, the differences in $\bar{x}$ values between the surface method and the extraction–pH meter method ranged from 0.2 to 1.55 units (white columns), whereas for water W_2 (green columns), the differences ranged from 0.3 to 1.35 units. These data indicate good convergence of the results of some of pairs of measurements using the surface and extraction-pH meter methods and significant divergence for some others. This can be partly likely due to the trend visible in Fig. 2, where surface method measurements tended to be lower than those obtained with the extraction–pH meter method. A similar tendency has been reported by Saverwyns *et al.* 2002; Strlič *et al.* 2004; Reist 2006; Blüher 2006; and Ahn *et al.* 2011, who noted that surface pH can be 1 to 2 pH units lower than the "effective" pH of the paper determined from an aqueous extract.

The data in Table 2 also allow a comparison of the convergence of pH measurements for papers from books B_1, B_2, and B_3 using two types of litmus papers and pH meter measurements of extracts from cold and hot extractions of these papers. For this purpose, the Δ_2 index was used. The values of this index for pH measurements of papers from Books B_1, B_2, and B_3 using water W_1 (black curves) and water W_2 (red curves) are presented in Fig. 4.

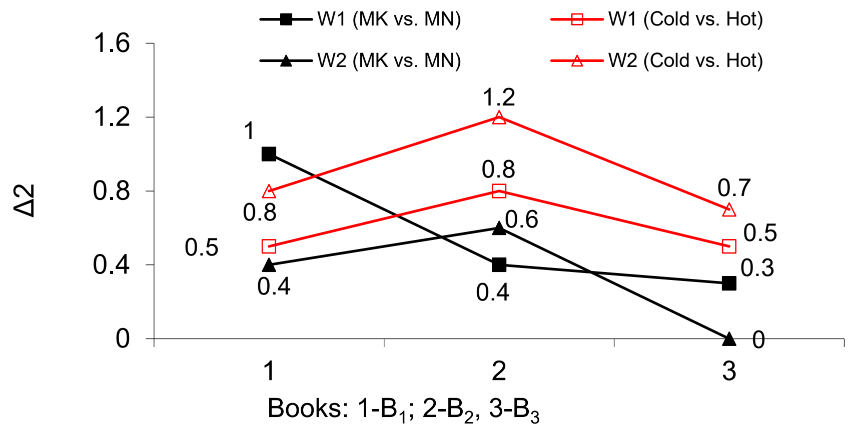


Fig. 4. Differences in pH measurement values of papers from books B_1, B_2, and B_3 using Merck and Macherey-Nagel litmus papers (black curves) and filtrates from cold and hot extractions of these paper samples (red curves)

As shown in Fig. 4, the pH measurements using MK and M-N litmus papers were relatively convergent. In four out of six cases, the differences between the mean values obtained with these litmus papers were less than 0.5 units. Only in two out of six measurements did the differences exceed 0.6 and 1.0 units, respectively.

In contrast, the pH measurements of papers from books B_1, B_2, and B_3 using the extraction–pH meter method were more divergent. In this case, two out of six measurements showed a difference of less than 0.5 units; three out of six differed by 0.7 to 0.8 units; and in one out of six cases, the difference reached 1.2 units. It appears that a significant factor contributing to the greater divergence in pH results obtained using the extraction–pH meter method is the large difference in extraction temperatures (cold *vs.* hot), which led to noticeably lower pH values in the hot extract. A similar pattern of clearly lower pH values in hot extractions compared to cold extractions has been observed by Barrow (1964) and Wilson and Hebert (1972).

The fact that, for some of the books selected for testing, the pH values obtained using both the surface and extraction - pH meter methods were sometimes convergent and in other cases divergent may also have been influenced by differences in the pH of the paper sheets in these books. The use of more than one pH method for papers from old books, newspapers and documents therefore has a positive impact on the ability to determine the actual degree of their acidification.

Yellowness of Paper

In 1981, Hunter reported that for approximately 100 years, the yellowness of materials had been assessed by visual comparison with either standardized glasses or standardized solutions of reagent-grade chemicals (*e.g.*, visual scales such as Lovibond, Hazen, Gardner, and FAC). Since 1942, paints, textiles, plastics, and many other materials have been evaluated for yellowness using photoelectric tristimulus scales (ASTM D1925, ASTM E313, Hunter b, and CIELAB *b**) based on the spectral response characteristics of the normal human observer and expressed as a percentage (yellowness) (Černič Letnat and Kropar Vančina 2002; Dragojević *et al.* 2021) or in numerical values (positive *b** parameter values) (Zervos and Moropoulou 2006; Fialová and Bartl 2025).

As described in the methodology, this study assessed the degree of yellowness using a visual (Y_V) evaluation conducted by the author and an instrumental method using the yellowness index measured with the Elrepho 2000 device (designated as Y_E). The use of the visual method for evaluating paper yellowness in conservation practice has been discussed by Winiarczyk and Rosa (2007), Sobucki and Czajka (2006), and Wagner *et al*. (2009), while the application of the Y_E index for this purpose was reported for example by Rams and Jarmińska (2006).

Regarding the visual method, the literature indicates that in conservation practice a three-level scale is often used, where Level I corresponds to no yellowing, Level II to slight yellowing at page edges, and Level III to complete yellowing (Sobucki and Czajka 2006). In this study, however, the scale proposed by Winiarczyk and Rosa (2007) was applied due to the uniform coloration of pages in books B_1, B_2, and B_3, the need to compare this degree with results from instrumental evaluation, and the observation that library holdings contain books with even higher levels of yellowing than book B_3. In this scale, 0 points correspond to no yellowing, 1 point to slight yellowing, 2 points to moderate yellowing, and 3 points to strong yellowing.

The results of assessing this paper characteristic using both methods are presented in Table 2. For comparison, the table also includes yellowness data for various types of book papers produced in the 1960s, as reported by Illukowicz (1968).

Table 2. Yellowness of Papers from Books B_1, B_2, and B_3 Determined by Visual (Y_V) and Instrumental (Y_E) Methods; The Index of a Contemporary Book Paper (CBP) and of Book B_3 After Two Years of Aging in Sunlight; The Visual Yellowness Scale; and the Leucometer Yellowness (Y_L) of Various Types of Writing and Printing Papers from the Late 1960s, as Reported by Illukowicz (1968)

Visual Method (M_1)	Y_V
B_1	1
B_2	2
B_3	2
Instrumental Method (M_2)	Y_E (%)
B_1	34.5 ± 3.7
B_2	50.9 ± 4.8
B_3	57.2 ± 2.8
Contemporary book paper (CBP)	-0.07 ± 0.02
Paper of book B_3 after two years' aging in sunlight	69.8 ± 2.5
Authors' yellowness scale based on Y_E: 0 point: 0-17.5%; 1 – points: 17.5-35.0%; 2 points: 35.0-52.5%; 3 points: 52.5-70.0%	
	Y_L
Writing/printing paper 1	3.0[1)]
Writing/printing paper 2	0.0[1)]
Writing/printing paper kl. III (a) (100% bleached chemical pulp[2)])	4.1[1)]
Writing/printing paper kl. III (b) (100% bleached chemical pulp[2)])	9.2[1)]
Writing/printing paper kl. III (a) (100% bleached chemical pulp[2)])	9.9[1)]
Writing/printing paper kl. III (b) (100% bleached chemical pulp[2)])	9.1[1)]
Writing/printing paper kl. V (40% GW and 60% bleached chemical pulp[2)])	22.9[1)]
Writing/printing paper kl. VII (70% GW and 30% bleached chemical pulp[2)])	24.8[1)]

[1)] Illukowicz 1968. Leucometer Carl-Zeiss-Jenna. Incandescent lamp. $Y_L = [R_{red}-R_{blue}/R_{blue}]\cdot 100\%$ (light reflectance with blue or red filter).

[2)] According to Wandelt; a, b – papers for the production of which the same pulp was used, but they were produced by other manufacturers

The data in Table 2 show that, based on visual assessment, the paper from book B_1 was assigned a yellowness level of 1. This evaluation indicated that the paper exhibited only a barely noticeable degree of this characteristic. However, when measured instrumentally using a colorimeter, the paper from book B_1 showed a Y_E value of 34.5%, which was 33.0% and 9.7% higher than the average yellowness of Writing Papers 1 and 2 and Class VII paper (with a fibrous, newspaper-type composition), respectively. These results suggest that within the 0 to 34.5% Y_E range, the perception of paper yellowness by the human eye may be only moderately objective.

For the papers from books B_2 and B_3, the Y_E mean values were 50.9% and 57.2%, respectively. When evaluated visually, these papers were considered moderately yellow (2 points on the visual scale). To verify the reliability of visual assessment of paper yellowness in books B_1–B_3, it was necessary to determine the possible range of variation of this characteristic. Table 2 indicates that the lower limit of yellowness for book papers is 0%.

Determining the upper limit of the yellowness scale is more challenging, as it was unclear whether approximately 50% yellowness represented the maximum value of this characteristic or if it could increase further. To partially address this issue, paper from book B_3 was artificially aged in sunlight for two years. The Y_E values of this paper are presented in Table 2. The data in Table 4 indicate that the maximum yellowness level of papers from old books can be assumed to be 69.8%, rounded to 70%. Accordingly, papers with Y_E values in the ranges 0 to 17.5%, 17.5 to 35%, 35 to 52.5%, and 52.5 to 70% should be assigned 0, 1, 2, and 3 points, respectively. Therefore, assigning 1, 2, and 2 points to the papers from books B_1, B_2, and B_3 based on visual assessment was correct for books B_1 and B_2 but incorrect for book B_3, which should have received 3 points for this characteristic.

Nevertheless, the convergence between visual and instrumental assessments of paper yellowness for books B_1, B_2, and B_3 can be considered good. It is difficult for the human eye to detect a yellowness difference of 57.2% to 52.5% = 4.7%, which led to the assignment of level 2 of yellowness to the paper from Book B_3 in the visual method, instead of level 3 as indicated by the instrumental measurement. It appears that the accuracy of visually assigning yellowness levels to papers from old books could be greatly improved by using reference standards of paper yellowness.

Weakening of Paper

This section focuses on evaluating the convergence and divergence of assessments of paper weakening in old books using methods such as the number of double folds (NDF) test (M_1), the tensile-after-six-folds (TSF) test (M_2), and the number of page turns (NPT) (M_3).

The first method (NDF) is a destructive test. As Fialová and Bartl (2025) stated, using this property as a strength test in conservation studies is justified because it is a compulsory test in the ISO 11108 standard, which specifies requirements for archival paper. For this reason, NDF is considered an important indicator of long-term paper durability. It has long been used in conservation research to determine the degree of weakening in old book papers (Zyska 1996) and to assess the influence of artificial aging on paper properties (Barrow 1964; Cardwell *et al*. 1972).

The second method (TSF) is used in Polish conservation practice for the rapid assessment of old book paper strength without destroying the material. According to Polish conservators, the test, which consists of folding one corner of a selected document six times and lightly stretching it, provides a reliable measure of the paper's functional strength. If the corner tears or detaches during the test, the paper is considered weak and in need of reinforcement (Sobucki and Czajka 2006). This test likely evolved from a combination of known conservation tests: brittleness and tensile-after-fold. Brittleness is defined as a paper's inability to undergo two double-fold tests (Mead and Baird 2003). In the brittleness test, the corner of a page is folded back and forth several times (one, two, three double folds, or more). If the corner breaks after one double fold, the paper is considered extremely brittle; after two double folds, it is considered brittle (Walker *et al.* 1985; Bond *et al*. 1987; Chrzastowski *et al.* 1989; Brown *et al.* 2020). In contrast, in the tensile-after-fold test, a paper strip is folded in a standardized manner: first, its two ends are fixed together on a plane at a 20° inclination, then pressed by a 460 g rolling cylinder, and finally, the tensile strength of the folded sample is measured using apparatus for measuring tensile strength (Bansa 1992; Lichtblau *et al.* 2008; Brown *et al*. 2020; Gmelch *et al*. 2024).

The third method (number of page turns — NPT) was devised by the author of this publication. It involves alternately turning a selected page of a book left and right, as one would when reading, until the first damage to the page are observed, and then continuing up to 1,000 page turns to visually assess the extent of damage to the tested page.

Table 3. Number of Double Folds (NDF) of Papers from Books B_1, B_2, and B_3 and of Contemporary Book Paper (CBP)

Kind of Book / Property	B_1	S.d.	B_2	S.d.	B_3	S.d.	CBP	S.d.
Basis weight of paper	90		70		60		90	
	NDF test							
MD (K-M[1])	11	3	1	-	1	-	873	121
CD (K-M)	29	2	1	-	1	-	504	86
MD (S)	17	6	1	-	1	-	1366	79
CD (S)	29	5	1	-	1	-	316	76
	TSF test							
Not applicable	Negative		Negative		Negative		Positive	

[1] K-M – property determined in Köhler-Molin apparatus
[2] S – property determined in Schopper apparatus

The results of assessing paper weakening using the NDF and TSF tests are presented in Table 4. The table shows that papers from books B_2 and B_3 exhibited very low, practically zero resistance to double folding when tested with the Köhler-Molin (K-M) and Schopper (S) devices, while the paper from book B_1 showed low resistance to this type of bending stress (11 to 29 folds). According to Zyska (1996), such papers can be classified as very weak or low-strength papers. These values corresponded to only 1.2 to 5.8% and 1.2 to 9.2% of the NDF test values for contemporary book paper (CBP) in the MD and CD directions, respectively (Table 4). In turn, as regards the results of determining the strength of paper using the TSF test, it indicated its very low level in the case of old books selected for testing.

As mentioned in the methodology, the strength of papers from old books was also assessed in this study using the NPT test, recording both the type of damage and the number of page turns required for that damage to occur. The results of these tests are summarized in Table 4, where **X** indicates a tear along the lower edge of the page, **Y** represents the detachment of a fragment from the lower edge (*e.g.*, approximately 5 mm wide and 2 to 3 mm high), **Z** denotes a tear at the point where the page folds during turning, *i.e.*, near the spine, and **0** indicates the absence of any **X**, **Y**, or **Z** type damages.

Table 4. Number of Page Turns of Three Pages from Books B_1, B_2, and B_3 until the First Damage Occurred (NPT) and the Type of Damage Observed

	B_1		B_2		B_3	
	Observed type of damage	NPT	Observed type of damage	NPT	Type of damage observed	NPT
Page 1	**0**	-	**X** (Fig. 3A)	750	**X** (Fig. 5A)	810
Page 2	**0**	-	**Y** (Fig. 3B)	420	**X** (Fig. 5B)	625
Page 3	**0**	-	**0**	-	**0**	-

As shown in Table 4 during 1,000 page turns of three initially undamaged pages from book B_1 no damage was recorded. In contrast, for books B_2 and B_3, **X**- and **Y**-type damages were observed in the case of Page 1 and Page 2. These damages were observed in two pages of book B_2 after 750 and 420 page turns, respectively, and in two pages of book B_3 after 810 and 625 page turns, respectively. No damage was observed for page P_3 in either book B_2 or B_3 after the NPT test.

Comparing the results of the NPT test with those of the NDF and TSF tests, it can be concluded that there is divergence between these methods. This is because none of the tested pages from books B_1, B_2, and B_3 experienced tearing along the left or right near-spine edges. Only tears along the lower edges of the pages or detachment of fragments from these edges occurred, and these appeared only after several hundred page turns in books B_2 and B_3 with a moderate severity (tearing of the edge of the page).

The extent of damage to the pages of books B_2 and B_3 in the NPT test is illustrated in Figures 5 to 8. Figures 5 and 7 depict the undamaged condition of selected pages from books B_2 and B_3, respectively, while Figs. 6 and 8 show their condition after several hundred page turns.

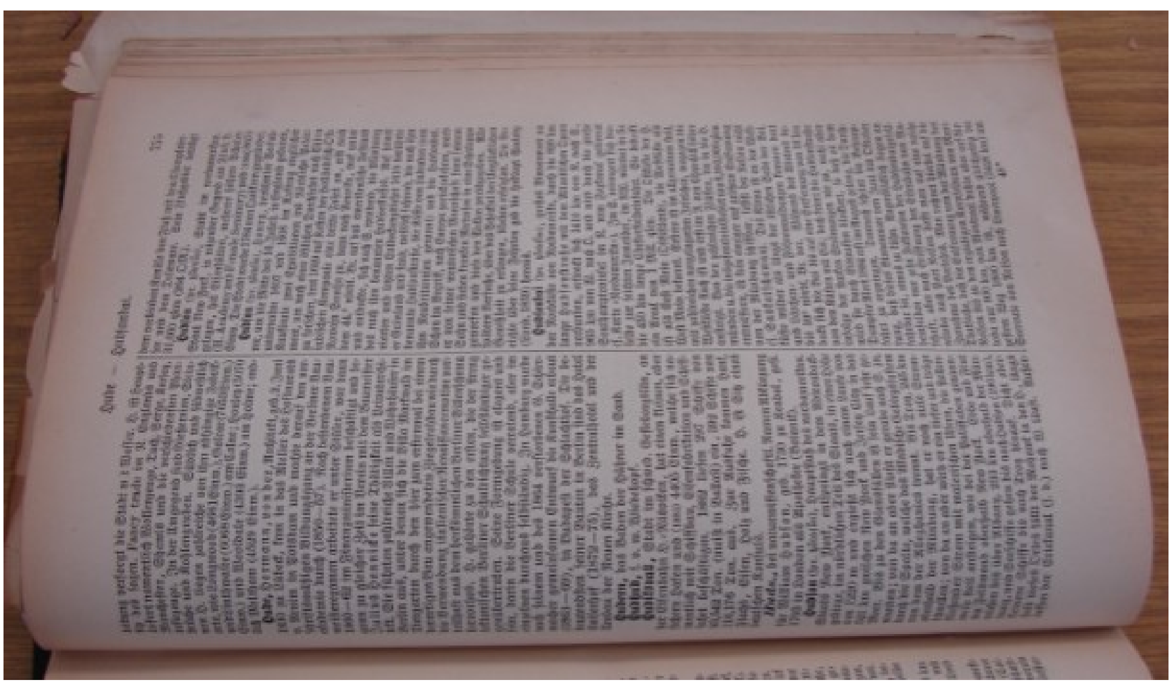


Fig. 5. Page of B_2 book before NPT test. No damage to the page is visible.

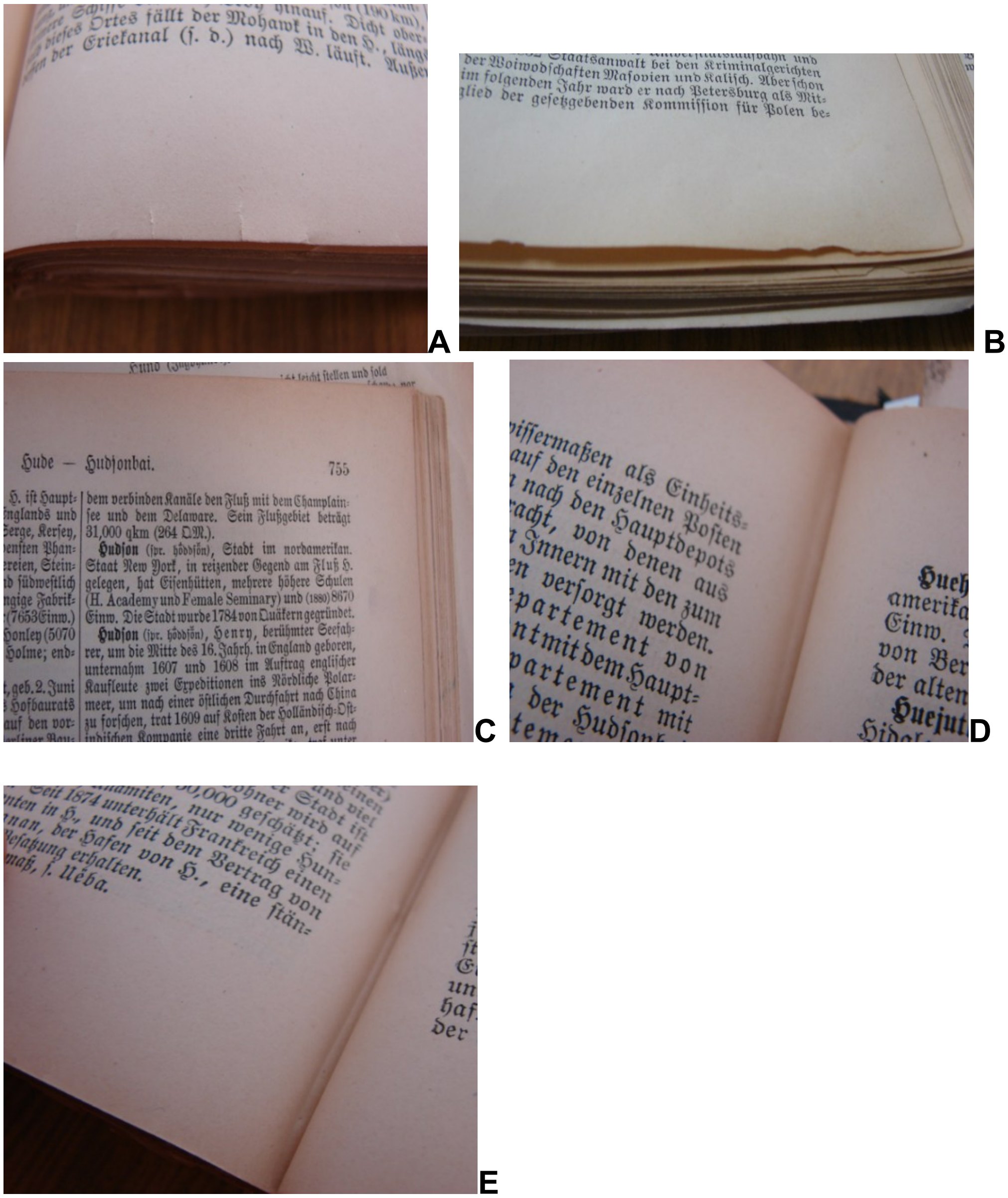


Fig. 6. Page 1 of Book B_2 after the NPT test. Visible are three tears along the lower edge of the page, up to 5 mm long (A); Page 2 of B_2 - three detachments of fragments from the lower edge (B); no tears along the upper edge of the page (C); Page 1, 2, and 3 had any damage to the spine-side of the page, with only impressions from repeated folding visible (D and E) after 1000 turns.

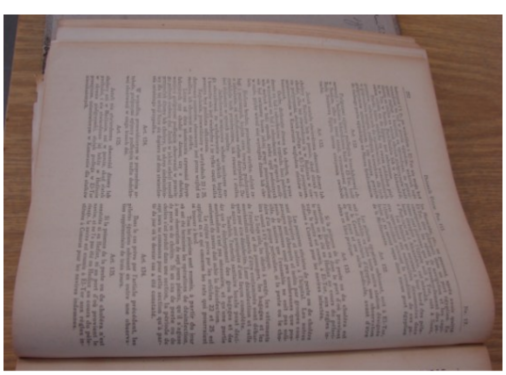


Fig. 7. Page of B_3 book before *NPT* test. No damage to the page is visible.

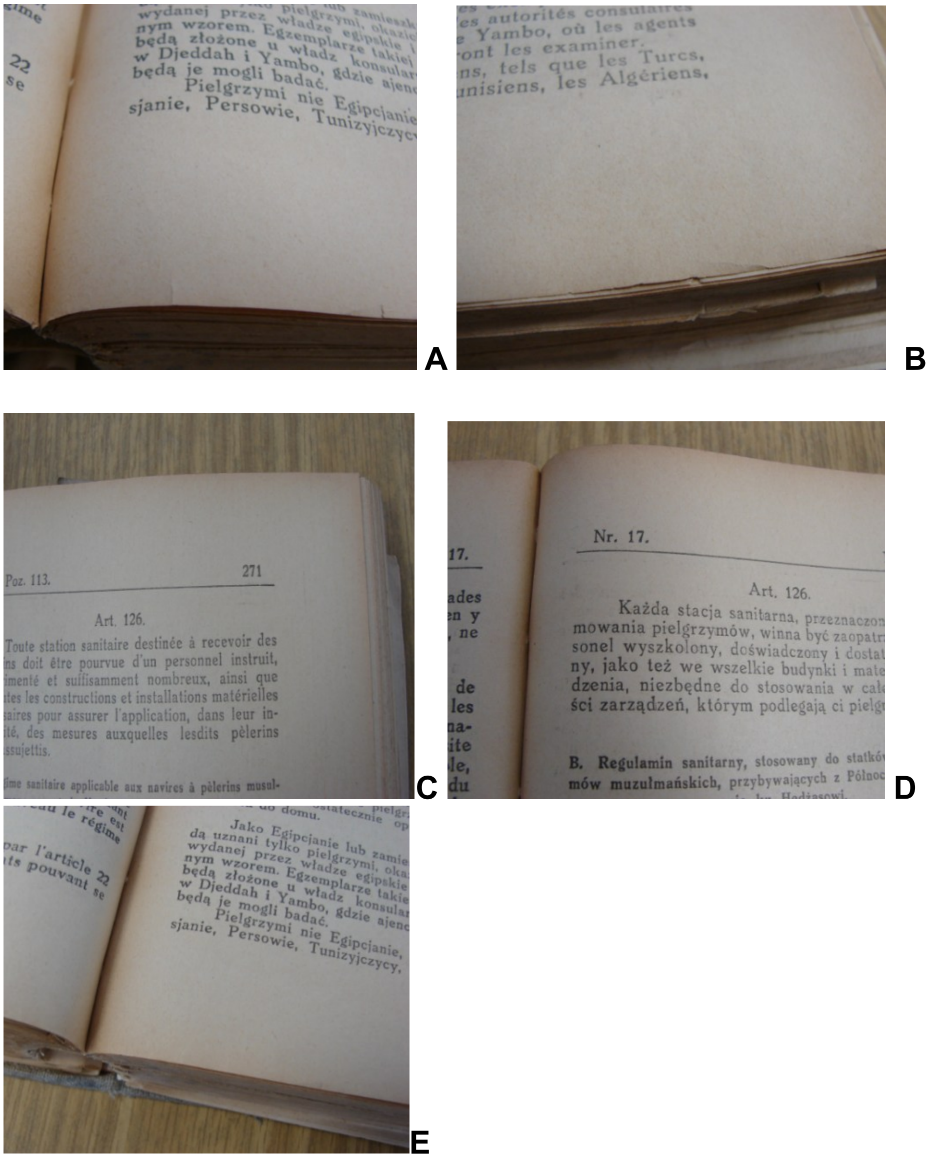


Fig. 8. Page 1 from Book B_3 after the NPT test. Visible are three tears along the left lower edge of the page, up to 5 mm in length (A); page 2 of Book 3 – two tears along the right lower edge (B); Page 1, 2, and 3 had no tears along the upper edge (C) or the side edges (D), and no damage to the spine-adjacent part of the page (E) after 1000 turns.

The considerable remaining strength of the papers from books B_1, B_2, and B_3 demonstrated in the NPT test was confirmed by their tear resistance measurements presented in Table 5, which corresponded to 47.2%, 32.0%, and 35.9% of the tear resistance of contemporary book paper (CBP), respectively.

Table 5. Tear Resistance of Papers from Books B_1, B_2, and B_3, and Contemporary Book Paper (CB)

Property	B_1	S.d.	B_2	S.d.	B_3	S.d.	CB paper	S.d.
Tear index. MD, mN□m^2/g	4.26	0.09	2.94	0.07	3.23	0.07	8.91	1.26
Tear index CD, mN□m^2/g	4.44	0.14	2.96	0.13	3.39	0.06	9.53	0.95

S.d. – standard deviation

The obtained results thus suggest the need to reconsider the applicability of the TSF and NDF tests for assessing the degree of paper weakening in old books. The likely low convergence between the assessment of paper weakening using the TSF and NPT tests arises from the negative effect of folding a page corner six times before applying tension in the TSF test, which significantly weakens even new paper, let alone paper from old books. Similarly, the low convergence between assessments using NDF and NPT likely stems from the much higher stresses imposed on the paper in the NDF test compared to the stresses experienced during careful page-turning by a reader. A second reason for the lack of convergence between NDF and NPT assessments may be the strong dependence of NDF test values on the paper's tensile strength, which in turn depends on the type of fibrous raw material used in its production and its degree of refinement.

To confirm this hypothesis, the types of fibrous raw materials used to produce the papers from books B_1, B_2, and B_3 were examined (Table 6) and in Figs. 9 to 11 literature data on the relationship between NDF and intrinsic tensile strength for straw, rag, cotton, groundwood, bleached kraft pulps from pine, birch, beech, eucalyptus, and sulfite spruce, as well as coated papers made from mixtures of some of these pulps was summarized.

From the data in Table 6, it appears that the paper of book B_1 (1928) was made from sulfite spruce pulp and rags. This was evidenced by the blue coloration of the fibers in Herzberg reagent, the low kappa number of the paper, and the presence of fibers characteristic of these raw materials in the microscopic images. The paper from book B_2 (1892) was likely made from a mixture of pulps from straw, spruce, and rags, and it also contained some groundwood. As for the paper from book B_3 (1933), studies showed that it contained a high proportion of groundwood fibers, which is also reflected in its relatively high kappa number. These were commonly used fibrous raw materials for printing papers between 1890 and 1935 (Kozielec 2011). Jabłońska (2007) reports that manuscripts from the Kórnik Library dated 1801 to 1870 contained almost exclusively rag pulps, while an analysis of fibrous composition in paper samples from 1871 to 1914 identified pulps including rag, straw, mechanical (groundwood), and, in a few cases, sulfite and kraft pulps.

Table 6. Fibrous Composition and Kappa Number of Papers from Books B_1, B_2, and B_3 and Contemporary Book Paper (CBP)

Paper Symbol	Kappa Number	Probable Type of Fiber Semi-Finished Product Used
B_1	8.3 ±0.3	Spruce pulp Long-fibered rag pulp
B_2	8.7 ±0.3	Straw pulp Spruce chemical pulp Rag pulp Groundwood
B_3	85.4 ±1.2	Groundwood Spruce chemical pulp
CBP	1.2	Bleached kraft hardwood pulp Bleached kraft softwood pulp

The data in Figure 9 support the hypothesis that straw, rag, and cotton pulps exhibit very low NDF values at low tensile strength (breaking length). However, the NDF values of these pulps can sometimes reach higher levels at greater breaking lengths (6 to 8 km), as in the case of straw pulp produced by the sulfite method (approximately 500 folds) and linters/rag pulps (approximately 1,000 folds), but only with appropriate selection of raw materials and processing conditions (*e.g.*, mild conditions for chemical pretreatment of rags). In contrast, the NDF values of groundwood pulp remain very low (a few double folds) or, at most, around 300 folds.

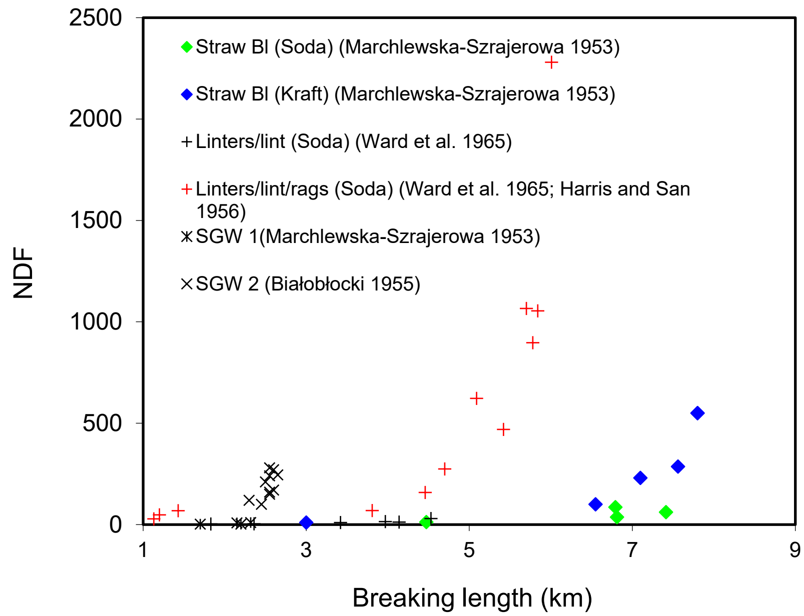


Fig. 9. NDF values of handsheets made from straw, rag, and cotton pulps, as well as groundwood, at specific breaking length (figure created by the author based on data from Białobłocki 1955, Marchlewska-Szrajerowa 1953, Ward *et al.* 1965, Harris and San 1956)

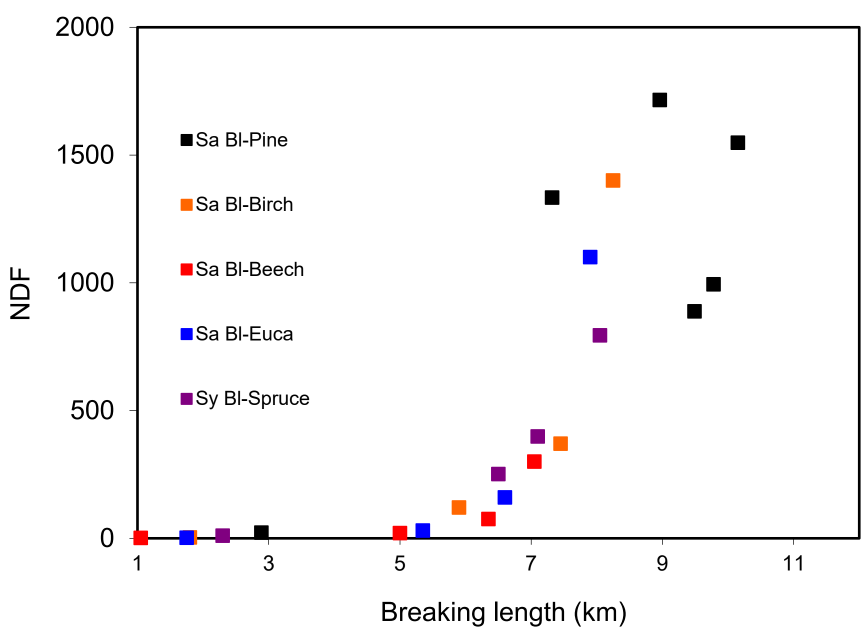


Fig. 10. Values of *NDF* of handsheets of bleached kraft (Sa) pulps from pine (Bl-Pine), birch (Bl-Birch), beech (Bl-beech), and eucalyptus (Bl-Euca) (figure created by the author based on base on tabular data presented by Hasvold and Lund 1974), as well as bleached spruce sulfite pulp (Bl-Spruce) at a specific value of their breaking strength (authors' work on the basis of numerical data presented by Fossum 1982)

In turn, the data in Fig. 10 show that a similar trend can be observed for bleached sulfite pulps, a very common intermediate material in book papers from the late 19th and early 20th centuries. NDF values of handsheets of this pulp remain low when the breaking length is low; however, at higher breaking lengths, The NDF values of such pulps increase. Interestingly, a similar trend can also be observed in bleached sulphate pulps derived from pine, birch, beech, and eucalyptus.

In the literature of the subject (Marchlewska-Szrajerowa 1953), NDF values are also reported for internally sized (but unfilled) writing (W) and printing (P) papers made from mixtures of straw pulp (Straw), groundwood (GWD), and rag pulp (Rags) in various proportions, as well as for such kind of papers made entirely from straw or groundwood (Fig. 11).

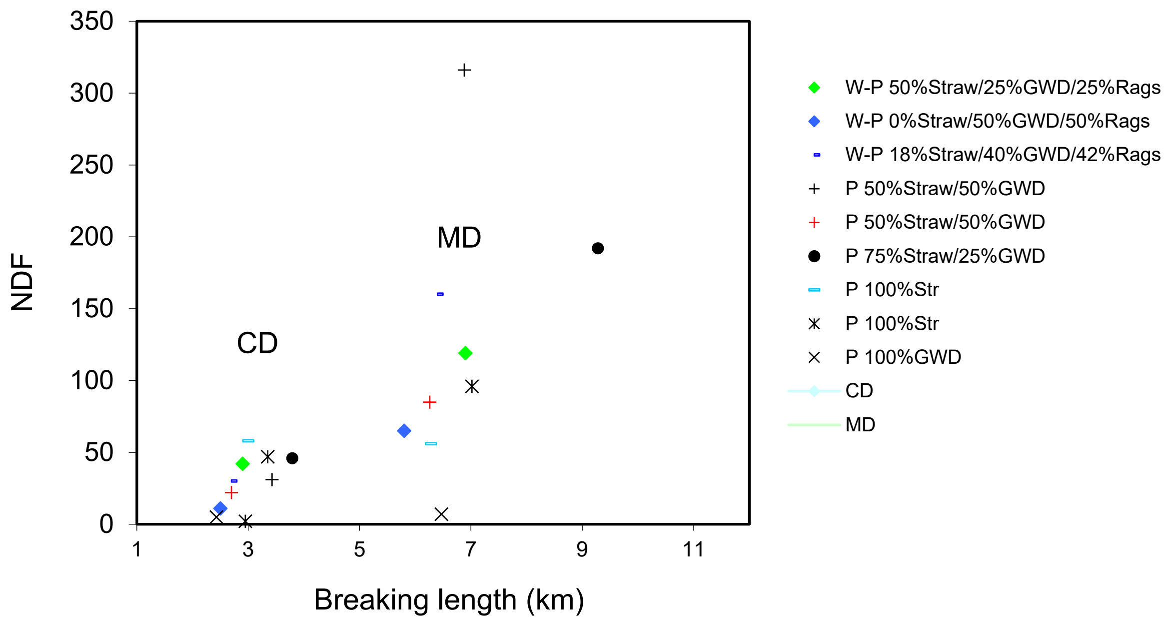


Fig. 11. *NDF* values of handsheets of writing (W) and printing (P) papers made of mixtures of straw pulp (Str), groundwood (GWD) and rag (Rags) pulps, and clean straw and groundwood pulps. (figure created by the author based on tabular data presented by Marchlewska-Szrajerowa 1953) (higher values MD, lower values CD)

As these data show, the NDF values in the MD direction ranged from 50 to 350 double folds, and in the CD direction from 0 to approximately 50 folds; thus, in both cases, they were relatively low considering the breaking length in the first case, which was 6 to 9 km (Marchlewska-Szrajerowa 1953). It is therefore not surprising that the papers from books B_1, B_2, and B_3, produced using rag pulp, groundwood, straw, and a certain amount of sulfite pulp with low tensile strength (22 to 30 N·m/g in the MD direction and 10 to 16 N·m/g in the CD direction) (Table 9), are characterized by low NDF values.

Table 9. Tensile Index of Papers from Books B_1, B_2 i B_3 and CB Paper

Property	B_1	S.d..	B_2	S.d.	B_3	S.d.	CBP	S.d.
Tensile index, MD	30.33	0.72	21.18	6.42	22.02	3.17	42.9	1.22
Tensile index, CD	15.73	1.54	13.09	5.89	10.38	2.87	22.4	6.3

MD – machine direction; CD – cross direction; S.d. – standard deviation

Taking into consideration the results of this study, together with the long history of the successful use of the NDF test to assess the effects of aging on paper strength, it seems that this test may not be a suitable method for assessing the degree of paper deterioration in books that have undergone prolonged natural aging and experienced a critical loss of mechanical strength or that were manufactured from papermaking pulps with inherently low tensile strength, resulting in its naturally low NDF values. Bansa (1992) also noted that the NDF test is too severe to provide meaningful data for weak papers undergoing conservation treatment.

CONCLUSIONS

1. Measurements of the overall pH of papers from three old books using the surface method (two types of litmus papers) and two extraction–pH meter methods generally showed good convergence. However, some results of pH measurements obtained using these methods were not always convergent. This discrepancy is likely due to the surface method yielding lower pH values than those obtained with the extraction–pH meter method. The pH measurements using the litmus papers were more convergent with each other than the pH measurements obtained from cold and hot extracts using the pH meter. This is probably because the hot extracts naturally had lower pH values than the cold extracts. The use of several pH measurement methods for paper from old books, newspapers, and documents has a positive impact on accurately determining their level of acidification.
2. Determination of the degree of yellowing of the papers from old books using the visual method and instrumental (the Elrepho 2000) method along with the yellowing scale showed good convergence for the papers from books B_1 and B_2, with a slight underestimation of the yellowness of book B_3. Research therefore indicates that it is possible with great probability to accurately estimate the yellowness of paper in old books using a visual method.
3. The assessment of paper weakening using the number of double folds (NDF) and tensile-after-six folds (TSF) tests showed good convergence but divergence when compared to the assessment based on the number of page turns test (NPT). The high degree of weakening of old books papers resulting from measurement made using the NDF test is likely largely due to the low tensile of these old papers at which NDF shows inherently very low values.
4. The high NPT values of pages from books B_1, B_2, and B_3 observed until minor damage occurred are likely due to the retention of tensile and tear strength in the MD and CD directions, yet amounting to approximately 46 to 71% of the tensile strength and 33 to 48% of the tear resistance of contemporary book paper.

5. Further research should be carried out to better explain the relatively small damage to book pages in the NPT test observed in this study (damage only along the lower edges of the pages, with no damage on the spin-side of pages even after its 1000 turns) with alarming results of the NDF and TSF tests. These observations indicate that the methods used to assess the state of preservation of papers from old books do not perfectly reproduce the stresses occurring when the reader turns the book pages, for example with regard to *e.g.*, a very short radius of bending in the NDF test compared to the much less sharp bending radius of paper in the case of the NPT test.

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Conflict of Interest

The results presented in this article were obtained by the author of this publication or with the assistance of his technical staff.

Use of Generative AI

The author did not use artificial intelligence in the preparation of this article.

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