Properties of lignocellulosic packaging materials for the long-term storage of diazotype records in depositories

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This study evaluates the suitability of selected lignocellulosic papers as packaging materials for the long-term preservation of diazotype records. To assess possible packaging materials, five types of paper were tested under artificial ageing conditions that simulate long-term storage. Their optical, chemical, and mechanical properties were systematically analysed.

The results revealed that two tested alkaline papers exhibited high optical and mechanical stability, alkaline pH values, and limited release of harmful volatile organic compounds. Whatman No. 1 unsized paper showed good optical stability and chemical neutrality. Two types of acidic paper sized with rosin performed poorly, releasing volatile acids and displaying low mechanical resistance. In general, both alkaline papers are recommended as packaging materials for the preservation of the diazotype, while Whatman No. 1 represents a possible compromise. Acidic papers are unsuitable because of their acidity, the emission of harmful degradation products, and their insufficient durability.

INTRODUCTION

The term diazotype (also called diazography) refers to copying techniques in which a paper or film is coated with a photosensitive layer containing diazo compounds. Due to its technical simplicity, low cost, and broad applicability, this process was widely adopted throughout the 20th century, although it has since been almost completely replaced by digital reproduction methods [1]. The principle of diazotype copying is based on two key chemical reactions: the coupling reaction of diazonium salts with amines or phenols to form azo dyes (Eq.1), and the photodecomposition of diazonium salts under ultraviolet radiation in the range of 350–420 nm (Eq. 2) [2].

$$[Ar1N_2]^+ X^- + Ar2 - OH \xrightarrow{pH>7} Ar1 - N = \\ = N - Ar2 - OH + HX \quad (1)$$

$$[Ar1N_2]^+ X^- + H_2O \xrightarrow{h\nu} Ar - OH + N_2 + HX \quad (2)$$

Under controlled alkaline conditions, exposure to ammonia vapours induces the coupling reaction, resulting in the formation of a positive image with characteristic colouration [3]. Several variations of the coupling reaction with amines or phenols exist. The most commonly applied and best-described procedure involves the incorporation of both the diazonium salt and the coupling components into the photosensitive layer. The pH of the paper is initially adjusted to prevent spontaneous reaction. Following exposure, the layer is subjected to ammonia vapours, which increase the pH and

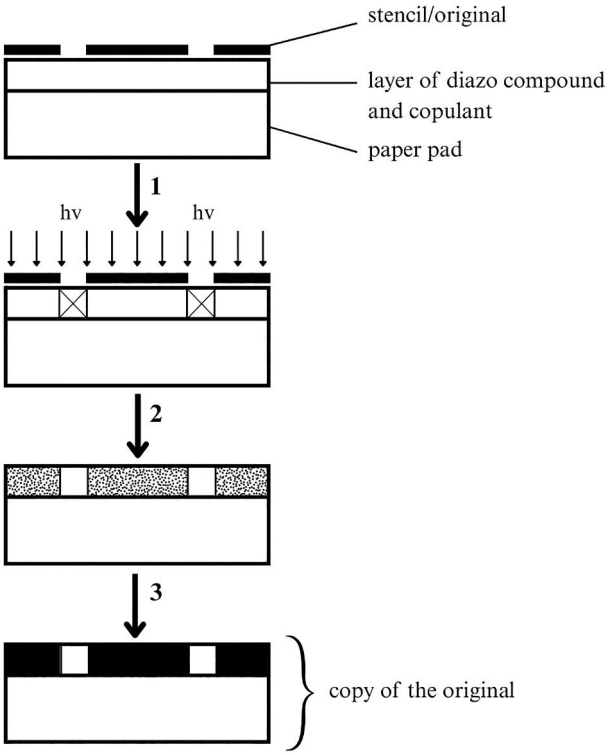


Fig. 1. Schematic representation of the diazographic process: 1 – exposure of the diazo compound layer to UV light, decomposition of the diazo compound in exposed areas; 2 – alkaline development, coupling (with ammonia vapours or alkaline developer); 3 – drying of the positive image, copy

enable the reaction, thereby producing a colour change in the unexposed areas. The result is the formation of a positive image. The process is schematically described in Fig. 1 [3].

The traditional substrate material for diazotypes is paper containing diazonium salts, primarily because of its availability and low cost. Such paper must be manufactured from high-quality raw materials, with an α-cellulose content of approximately 95%. Although cotton represents an ideal but economically expensive source of fibers, in practice, sulfite pulps from beech, spruce, and aspen were preferred. In the Czech Republic, diazographic materials were produced by Repro, s.p. Český Brod [4,5].

The lifetime of diazographic records is very limited, typically ranging from four to eight months after production. This period could be extended by storage under cold conditions (5 °C or below). Another essential storage requirement was the prevention of contact with ammonia residues or other reactive chemicals that could interact with the surface of the material [5].

Light sensitivity poses a fundamental problem for diazotypes, as the image formed after exposure to ultraviolet radiation gradually fades and may eventually disappear completely (Fig. 2). Direct sunlight not only causes fading but also yellowing, especially in lighter areas. Comparable colour changes may also occur during prolonged exposure in micrographic readers [5]. The degradation process is further influenced by the manufacturing technology itself and is often accompanied by a characteristic, intense odour. Although these degradation mechanisms have not yet been described in detail, it can be assumed that residual components from the chemical copying process continue to react with the paper substrate. The phenols contained in the chemical reagents are particularly prone to oxidation, which in turn affects the overall colour stability of the diazotypes [6].

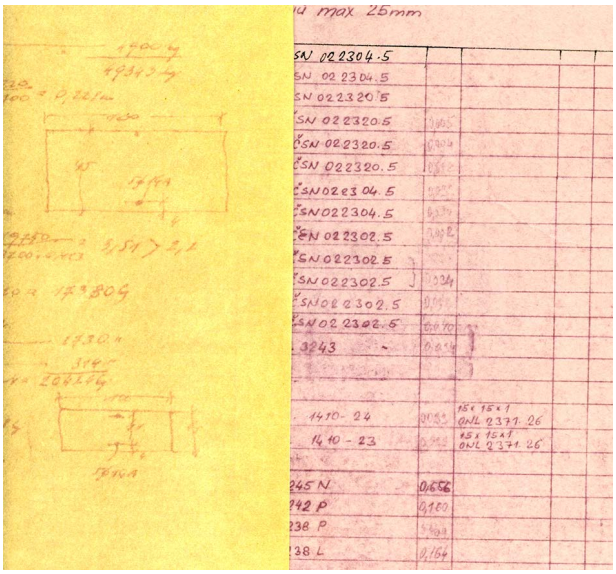


Fig. 2. Fading and disappearance of diazotype records

For long-term storage of diazotype records in depositories, appropriate packaging materials play a critical role in addition to climatic conditions. Preference is given to cellulose- or lignocellulose-based materials. For light-sensitive photographic materials, chemically inert packaging systems are recommended, which must comply with the requirements of ISO 18902:2001. This standard specifies that the packaging paper should contain at least 87% α-cellulose, be free of lignin, dyes, UV absorbers, and rosin-based sizing agents, and be neutrally sized with alkyl ketene dimers. The pH of such papers should range from 6 to 7, and the content of reducible sulfur should be below $2 \cdot 10^{-4}$ wt.%. In addition to these compositional requirements, the Photographic Activity Test (PAT) is sometimes used for evaluating packaging materials for photographic records [7]. However, no such specific requirements have yet been established for diazotype packaging.

For this reason, the present study focused on a detailed investigation of the properties and resistance to artificial ageing of selected lignocellulosic materials, with the aim of assessing their potential use as packaging materials for the long-term preservation of diazotype records.

MATERIALS AND METHODS

Tested Materials

Five types of paper were tested. An overview of the papers tested and their basic properties is given in Tab. 1. All papers were analyzed before and after artificial ageing, which simulated long-term storage conditions.

Tab. 1 Characterisation of the papers tested

Paper type	Grammage [g/m²]	Thickness [mm]	Designation	Type of paper sizing
Document paper according to ČSN ISO 9706	80	0.009	ISO	alkyl ketene dimers (AKDs)
Alkaline paper	146	0.020	AP	alkyl ketene dimers (AKDs)
Paper for testing deacidification processes	70	0.009	TP	rosin size
Whatman No. 1 chromatographic paper	87	0.017	W1	unsized
Wood-containing paper with high groundwood content	52	0.012	DP	rosin size

Artificial ageing

The paper samples were subjected to two types of artificial ageing:

- Artificial ageing with moist heat (80 °C, 65% RH, 30 days) according to the ČSN ISO 5630/3 standard (labeled as "ageing 80").
- Artificial ageing with dry heat (105 °C, 12 days) according to the ČSN ISO 5630/1 standard (labelled as "ageing 105").

Moisture-treated ageing took place in a Memmert HCP 108 humidity chamber. Dry heat ageing took place in a Memmert UF 55 drying oven.

Optical properties

The ISO brightness of the paper samples before and after ageing was measured using a Leukometer (Zeiss Jena, Germany) at a wavelength of 457 ± 2 nm. The brightness B is defined according to Eq. 3, where R_∞ represents the reflectance of the measured surface and R_∞^0 is the reflectance of the brightness standard. To ensure opacity of the measured paper, a sufficiently thick layer was prepared by backing with paper of the same type.

$$B = \frac{R_\infty}{R_\infty^0} \cdot 100\ \% \tag{3}$$

Colour was evaluated spectrophotometrically (Konica Minolta CM-700d, SCI mode) in the CIE Lab* colour space. The total color difference ΔE* was calculated according to Eq. 4, where L_0^*, a_0^*, b_0^* are the color space coordinates before artificial ageing and L_1^*, a_1^*, b_1^* are the coordinates measured after artificial ageing. Nine identical positions were measured on each paper sample. From obtained data, the mean value and standard deviation were calculated.

$$\Delta E^* = \sqrt{(L_1^* - L_0^*)^2 + (a_1^* - a_0^*)^2 + (b_1^* - b_0^*)^2} \tag{4}$$

Chemical properties

Fibre composition

The fibre composition was determined according to ČSN ISO 9184 using the Herzberg (ČSN ISO 9184-3) and Graff "C" (ČSN ISO 9184-4) staining tests. Moistened paper samples were disintegrated with a scalpel and applied onto microscope slides, where they were dried and stained with the respective reagents. The stained preparations were observed under a light microscope (Olympus BX60) in transmitted light.

Degree of sizing

The degree of sizing was determined using the line method with a Noll pen (Jupiter 27-70-1) according to ČSN 50 0331. The degree of sizing represents the maximum width of the ink line that does not penetrate to the opposite side of the sheet. The ink line width is adjustable in increments of 0.2 mm increments within the range of 0.2 to 1.2 mm. On each paper tested, six parallel lines were drawn on each side of the sheet. From the results, the arithmetic mean and standard deviation were calculated. Paper W1 was not tested, as no sizing agents are used in its production.

Determination of the pH of the cold aqueous extract

The measurement was carried out according to ČSN ISO 6588. The paper samples were extracted with demineralised water (conductivity 0.06 µS/cm). The pH value of the cold extract was determined using a WTW 7310 pH meter with a glass pH electrode (WTW Sentix 41). For each sample, two measurements were made and the arithmetic mean was calculated.

Cross-infection test

To assess the influence of volatile compounds released from the samples on cellulose, a cross-infection test was performed according to the literature [8]. The paper samples were aged in hermetically sealed bottles together with Whatman No. 1 paper at 80 °C for 14 days. After ageing, the average degree of polymerisation (DPη) of the Whatman No. 1 paper was determined according to ISO 5351-1. The limit viscosity number [η] was measured using an Ubbelohde capillary viscometer. For each sample, two measurements were made and the average degree of polymerisation was calculated according to Eq 5. The DPη value of the independently aged Whatman No. 1 paper was used as a reference.

$$DP^{0.85} = 1.1 \cdot [\eta] \tag{5}$$

Analysis of VOC emissions

Samples of the tested papers that had not aged were cut into strips measuring approximately 0.5 × 5 cm. A quantity of 1.6 g of each paper was placed in special quartz ampoules (see Fig. 3) equipped with a capillary for the entry of SPME fibre. To remove adsorbed volatile

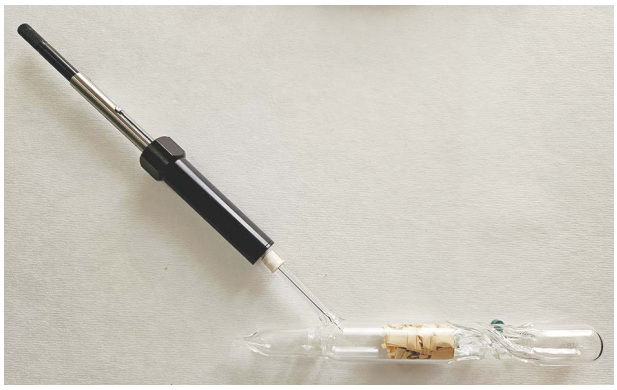


Fig. 3. Extraction of VOCs using a DVB/CAR/PDMS SPME fibre

substances from the environment, the ampoules with samples were heated for 1 hour at 60 °C. The ampoules were then hermetically sealed by sealing and placed in a drying oven (Memmert UF55) at 80 °C for 7 days. The extraction of VOCs was performed using a DVB/CAR/PDMS SPME fibre (50/30 µm, Supelco, 57298-U). The absorption times were 60 min at 23 °C (Fig. 3). A gas chromatograph (TRACE GC ULTRA, Thermo Scientific) coupled to an ISQ mass spectrometer (Thermo Scientific) was used to recover and analyse the substances from the fibre. The injector temperature was 250 °C and the injector was used in a split mode of 1:10. The carrier gas was helium 6.0 with a constant flow rate of 1.5 mL/min. A DB-5MS UI column (60 m × 0.32 mm, film 1.0 µm) was used for the separation of VOCs using a temperature programme as follows: starting temperature of 40 °C (hold for 5 min), a constant temperature increase at a rate of 15 °C/min until the final temperature of 290 °C (hold for 5 min).

Mass spectra were collected in the electron ionisation (EI) mode at 70 eV in the range of 29 to 450 Da. The scan time was 0.25 s. The VOCs peaks were identified using the NIST 20 database of MS spectra.

Mechanical properties

For the analysis of mechanical properties, the machine direction of the papers was determined and is further referred to as MD. Testing was performed both in the machine direction (MD) and in the cross direction (CD, perpendicular to the machine direction). Before testing, the paper samples were conditioned for 24 h at T = 23 ± 1 °C and RH = 50 ± 2%.

Tensile test

Samples of aged and unaged paper measuring 15 × × 120 mm were tested for tensile strength in both the MD and CD directions. A universal tensile testing machine Alwetron AH-1 (Lorentzen & Wettre, Sweden) was used for testing. The starting distance of the pneumatic jaws was 50 mm. Ten samples were tested for each type of paper. The results were used to calculate the tensile strength, the breaking length, the elongation at the break, and the absorption of the tensile energy. For each parameter, the resistance to ageing S was calculated according to Eq. 6, where Xs is the value of the monitored property of the sample after artificial ageing (e.g. tensile strength) and Xn is the value of the monitored property of the unaged sample.

$$S = \left(\frac{X_s}{X_n}\right) \cdot 100\ \% \qquad (6)$$

Zero-span test

The tensile strength at zero span was measured for aged and unaged paper samples in the MD and CD directions. For each paper, 10 pieces measuring 15 × 100 mm were tested using a Pulmac Z-Span 1200 Tester. The resistance to ageing was calculated from the measured values according to Eq. 6.

RESULTS AND DISCUSSION

Optical properties

Fig. 4 shows the brightness results for paper samples aged with dry heat (105 °C) and moist heat (80 °C, 65% RH). Dry heat caused only a slight decrease in brightness in all cases. A more significant decrease occurred after ageing with moist heat. The largest decrease was observed in ISO paper (approx. 24% decrease), while W1 paper showed the lowest change in brightness compared to unaged paper (approx. 16% decrease).

In the case of total colour difference measurements, samples aged with moist heat also achieved higher values (Fig. 5). DP and ISO papers showed the highest color change after moist ageing. In contrast, the lowest colour changes for both wet and dry ageing were measured for W1 paper. The main contributions to the total colour difference for all papers were a shift along the *L* axis to negative values and a shift along the b axis to positive values. It can therefore be said that accelerated ageing causes the tested papers to darken and yellow.

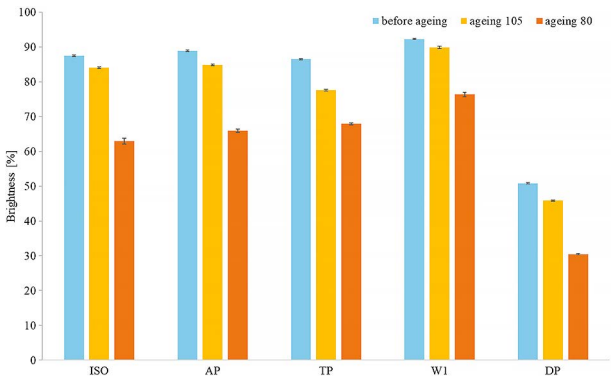


Fig. 4. Results of paper brightness measurements after artificial ageing

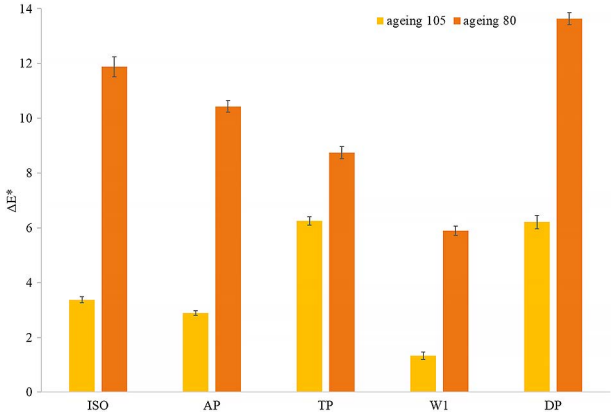
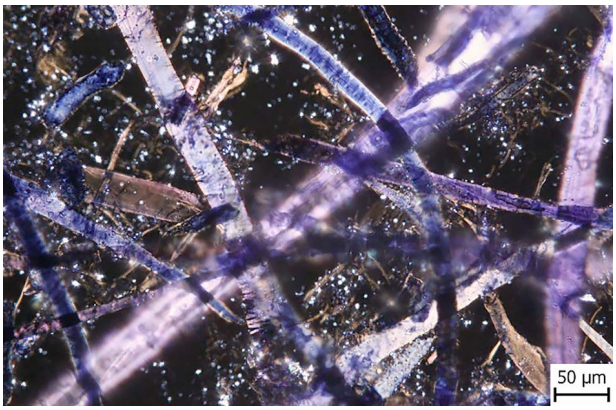


Fig. 5. Total colour difference of the papers after artificial ageing

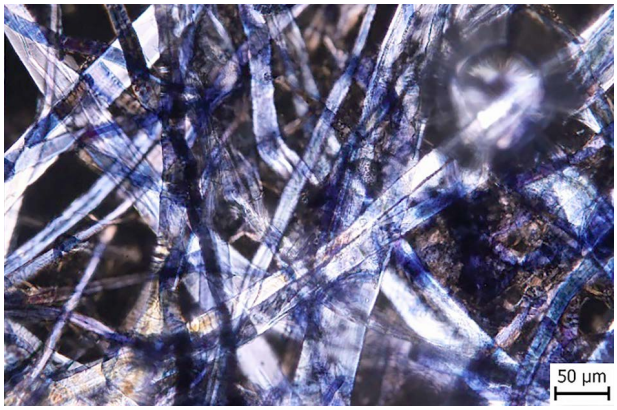
Results of chemical properties

Fibre composition

Figures. 6-10 show the results of staining tests on fibres taken from individual papers. The ISO, AP, and TP papers exibited the same colour reaction to the colouring agents. These papers contain bleached sulphate pulp derived from deciduous trees. Based on the colour reaction of the W1 paper (see Fig. 9), it indicates that the fibres of this paper are made of sulfite pulp derived from coniferous trees. The fibres of the DP paper were made of sulphate pulp from deciduous trees, as in the case of the ISO, AP, and TP papers (Fig. 6-8). However, in addition, the presence of wood pulp was identified, which is likely to have lower chemical stability depending on the production process [9].

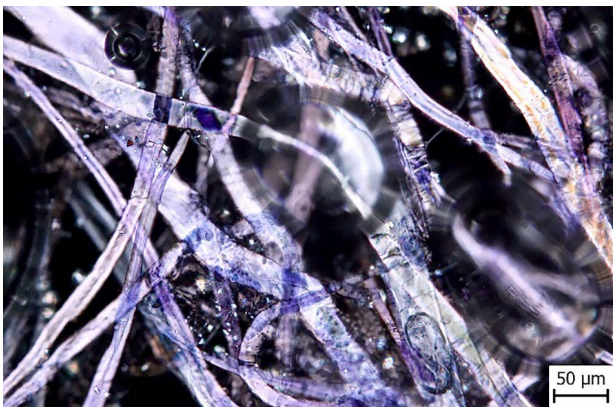


a) Herzberg test

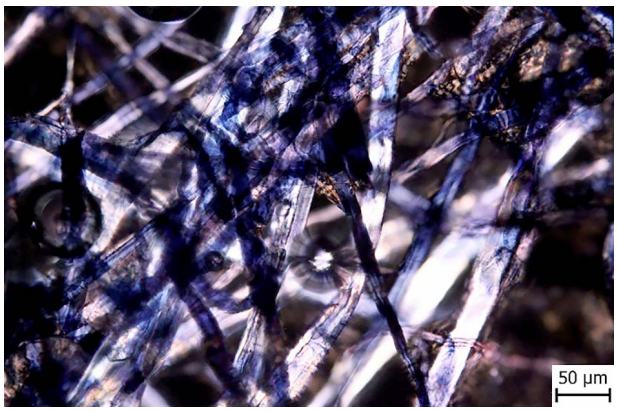


b) Graff C test

Fig. 6. Results of ISO paper staining tests

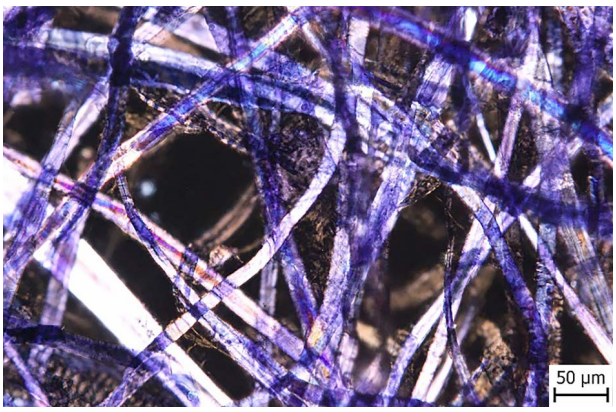


a) Herzberg test

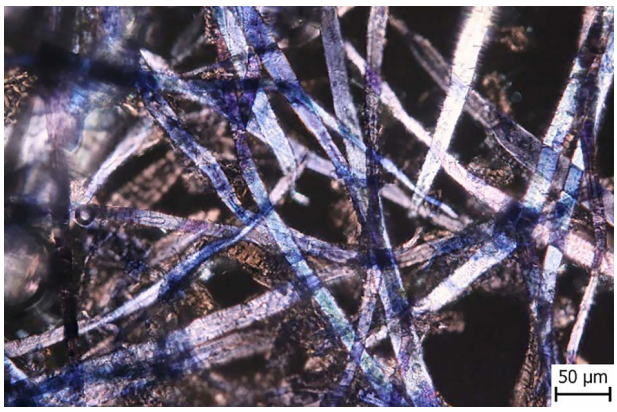


b) Graff C test

Fig. 7. Results of the AP paper staining tests

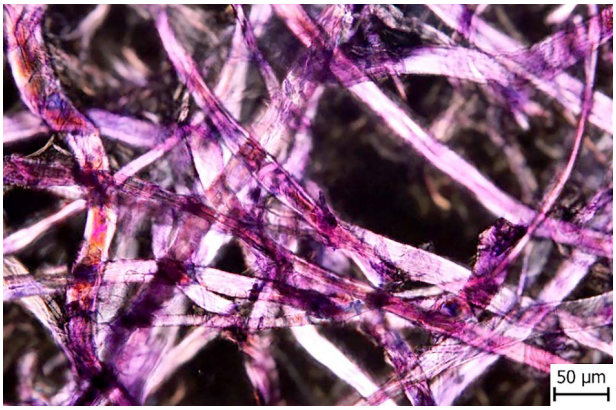


a) Herzberg test



b) Graff C test

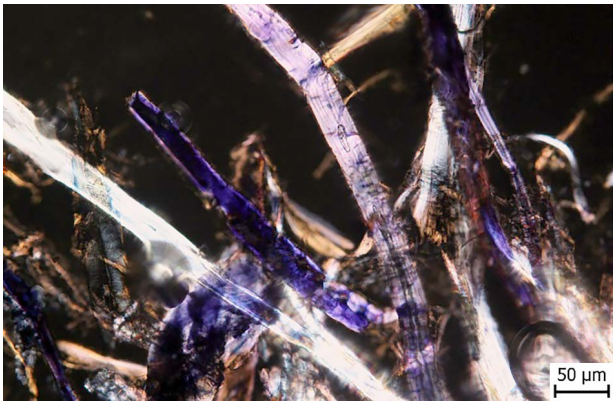
Fig. 8. Results of the TP paper staining tests



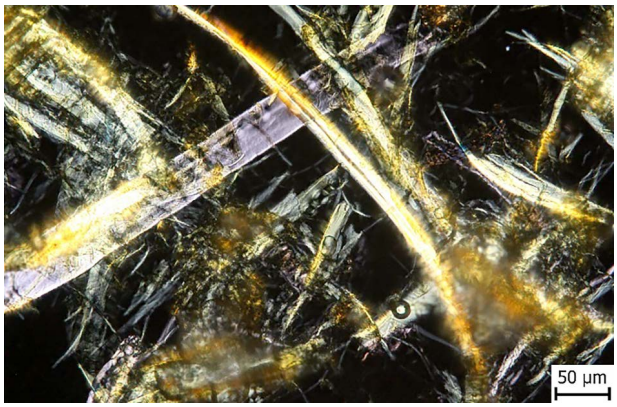
a) Herzberg test

b) Graff C test

Fig. 9. Results of the W1 paper staining tests



a) Herzberg test



b) Graff C test

Fig. 10. Results of the DP paper staining tests

Degree of sizing

Tab. 2 shows the results of the degree of sizing for the tested papers. The highest degree of sizing was achieved by ISO and AP papers. A lower degree of sizing was measured for TP and DP papers. W1 paper was not tested because no sizing agents are used in its production.

Tab. 2 Degree of sizing of tested papers

Paper type	Degree of sizing [mm]
ISO	1.2
AP	1.2
TP	0.35
DP	0.55

pH of cold aqueous extract

The results of the change in pH of the cold aqueous extract after ageing with dry and moist heat are shown in Fig. 11. Before ageing, ISO and AP papers showed a pH of around 9. After ageing in moist heat, the pH decreased to around 6.5. The W1 paper also showed a decrease in pH after ageing. After moist ageing, there was a decrease of 1.3 pH units. After dry heat ageing, the pH decreases in ISO, AP, and W1 papers were approximately half that after moist heat ageing. The decrease in pH is likely related to the formation of organic acids as a result of cellulose oxidation, the residual presence of hemicellulose and lignin, or, for example, the content of residual substances in the production of paper [10].

After artificial ageing, the TP and DP papers showed a slight increase in the pH values of the extracts. The pH value before ageing for these two papers was 4.2, which

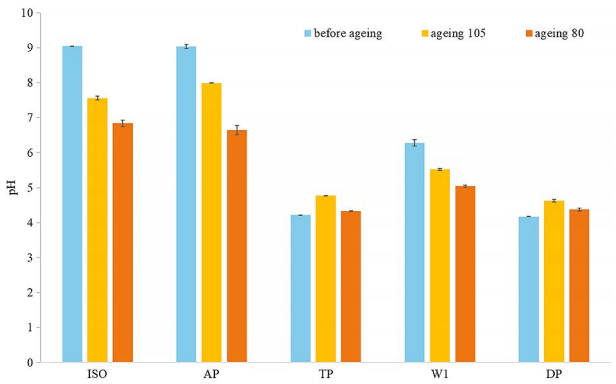


Fig. 11. Changes in the pH of cold aqueous extract after artificial ageing

is a relatively low starting value and is related to the pre. sence of acidic substances in the papers. The increase in pH after ageing may be caused by the leaching of acidic substances from the paper under the influence of increased temperature.

Results of the cross-infection test

The average polymerisation degree values of Whatman No. 1 paper after the cross-infection test, which quantifies the effect of VOCs released from the tested material on the paper, are shown in Fig. 12. VOCs released during the ageing of ISO, AP, and W1 papers had a comparable effect on reducing the DPη of cellulose. A greater effect, manifested by a more pronounced reduction in DPη, was observed in the TP paper and most notably in the DP paper. The low initial pH of these papers suggests that acidic compounds are released that attack the glycosidic bonds of the cellulose chain through acid hydrolysis [11].

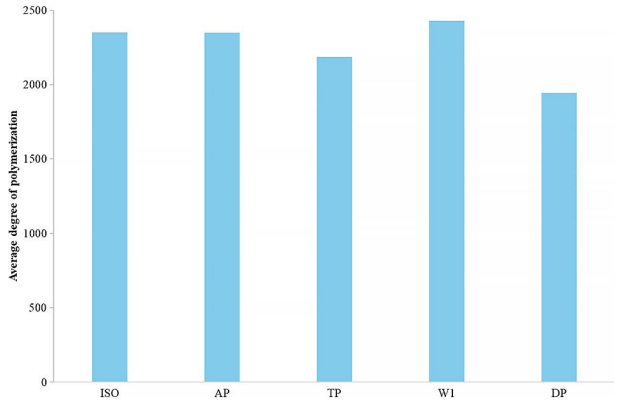


Fig. 12. Average degree of polymerisation of Whatman No. 1 paper after cross-infection testing

VOCs emission analysis

Fig. 13 shows chromatograms of desorbed substances from SPME fibres for individual papers, with designated substances marked as main peaks. The BLANK result represents a chromatogram of an SPME fibre placed freely in the laboratory to exclude the influence of contamination of samples by the surrounding environment. The chromatograms for ISO, W, and AP papers are very similar. The main substances released include acetone, isopropyl alcohol, 2,3-butanediol, furan, *n*-pentanal, and n-hexanal. In contrast, TP and DP papers released additional noticeable amounts of furfural. Furan cycle aldehydes are formed by the so-called β-elimination of a hydroxyl or alkoxyl group in the β-position relative to the carbonyl group [12-14].

In addition, substances based on the terpenes longicyclene and longifolene were detected in DP paper, probably originating from the wood pulp present, specifically from certain species of pine [15] (see results of Fibre composition, section 3.2.1).

To determine the visibility of possible organic acid release from the papers, the chromatograms were limited to the results for particles with a m/z ratio of 60 to 61. The result is shown in Fig. 14, where the presence of acetic acid can be reliably detected for TP and DP papers. The release of acetic acid, which can cause acid hydrolysis of cellulose, could explain the lower average degree of polymerisation after the cross-infection test (see Fig. 12). The release of acetic acid from paper is related to its composition and susceptibility to degradation. The wood content, or rather lignin, in DP paper contributes to the formation of organic acids through its oxidation [16].

Tab. 3 Ageing resistance values in tensile testing

		Ageing 105				Ageing 80			
Paper type	**Direction**	**Tensile strength [%]**	**Breaking length [%]**	**Elongation at break [%]**	**Tensile energy absorption [%]**	**Tensile strength [%]**	**Breaking length [%]**	**Elongation at break [%]**	**Tensile energy absorption [%]**
ISO	MD	97.6	97.6	90.2	85.6	97.1	97.1	90.2	87.8
	CD	97.0	97.0	89.1	88.0	97.5	97.4	82.7	80.9
AP	MD	109.2	109.1	89.9	97.2	107.3	107.2	86.3	90.8
	CD	102.8	102.8	70.2	71.7	104.7	105.2	84.4	54.1
TP	MD	63.0	63.2	64.4	41.1	88.0	88.1	82.0	67.3
	CD	96.2	95.3	50.8	43.5	80.3	79.8	72.7	57.3
W1	MD	97.5	97.5	88.7	85.2	92.1	92.6	85.3	76.0
	CD	106.3	106.2	94.8	100.8	92.2	92.4	90.5	81.1
DP	MD	111.5	111.2	98.6	96.0	40.4	40.4	107.5	35.6
	CD	95.5	95.4	88.7	74.3	77.4	77.8	70.0	43.5

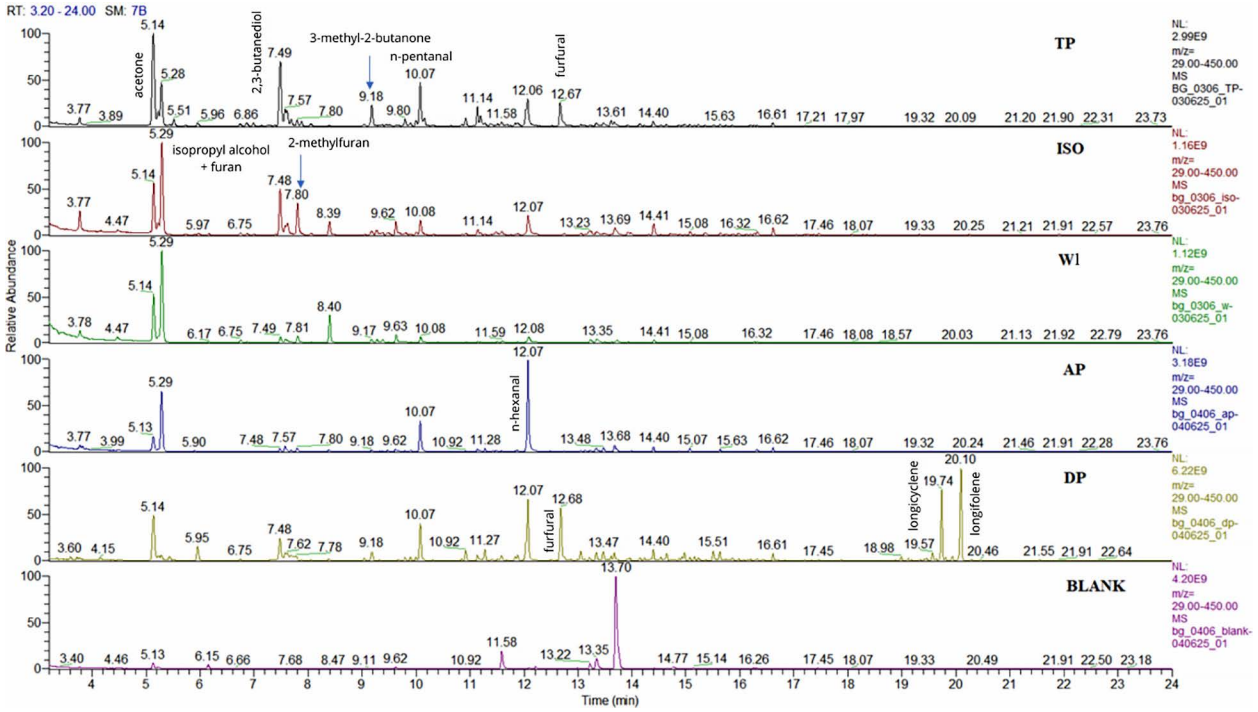


Fig. 13. Chromatograms of compounds desorbed from SPME fibres

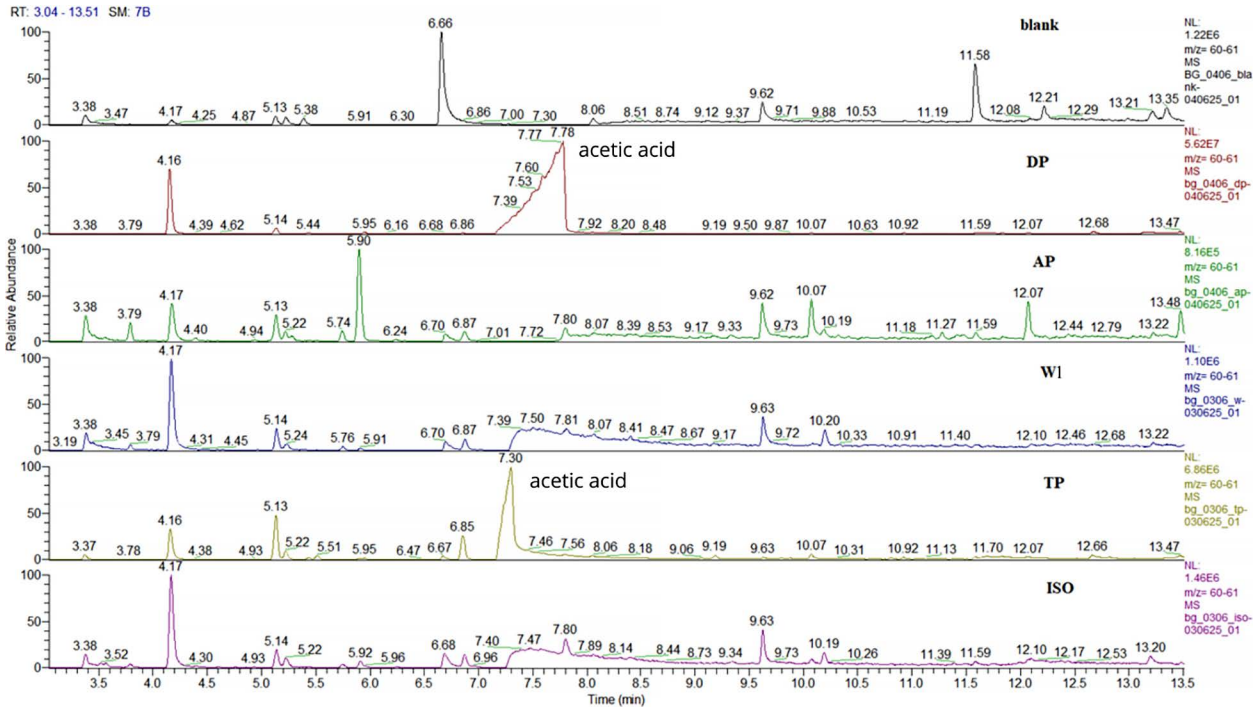


Fig. 14. Chromatograms of compounds desorbed from SPME fibres with restriction to m/z = 60–61

Results of mechanical properties

Tensile strength

Based on the four monitored parameters, the ageing resistance values were determined (Tab. 3). Values exceeding 100%, marked in gray in the table, indicate an increase in mechanical properties after artificial ageing. This effect was observed in AP paper (tensile strength and tear length in both directions under both ageing regimes), as well as in W1 paper (tensile strength, tear length, and tensile energy absorption in the transverse direction during ageing at 105 °C), and DP paper (tensile strength and tear length in the longitudinal direction during ageing at 105 °C and elongation in the longitudinal

direction during ageing at 80 °C). The observed increase in resistance can probably be explained by the cross-linking of bonds in the cellulose structure during ageing. In contrast, a systematic decrease in all evaluated properties was observed for TP paper. A significant deterioration was also observed for DP paper, with the exception of elongation in the longitudinal direction.

Tab. 4 Ageing resistance values in tensile testing at zero test specimen length

Paper type	Direction	Ageing 105	Ageing 80
ISO	MD	92.3	92.6
	CD	94.9	93.6
AP	MD	95.3	87.1
	CD	98.4	96.4
TP	MD	62.9	50.1
	CD	61.2	57.8
W1	MD	84.6	87.1
	CD	85.3	96.3
DP	MD	69.2	88.6
	CD	70.4	86.4

Zero span tensile strength

The results of ageing resistance (Tab. 4) show that ISO, AP, and W1 papers exhibited relatively low decreases in strength after both types of artificial ageing. In contrast, DP paper was shown to be less resistant to ageing at 105 °C, while TP paper showed a more significant loss of mechanical properties during ageing at 80 °C.

CONCLUSIONS

The analyses of optical, chemical, and mechanical properties revealed significant differences in the resistance of the tested paper types to artificial ageing and in their suitability for use as packaging materials for diazotypes. Whatman No. 1 paper proved to be the most optically stable, showing minimal brightness and colour change. In contrast, ISO paper, and especially DP and TP papers, exhibited stronger decreases in brightness or colour stability. ISO and AP papers exhibited an alkaline character and higher degree of sizing, while TP and DP papers were acidic with lower sizing. The cross-infection test and the VOC emission analysis confirmed that TP and DP release volatile acidic products (e.g., acetic acid) accelerating cellulose degradation. Mechanical tests revealed that ISO, AP, and to some extent, W1 showed good resistance to ageing, with AP achieving the best results.

In general, AP and ISO are the most suitable packaging materials for diazotypes, combining optical, chemical, and mechanical stability. Whatman No. 1 paper may serve as a reasonable compromise, offering high optical stability and chemical neutrality but limited strength. In contrast, TP and DP papers are unsuitable because of their acidic character, the release of degradation products, and poor mechanical stability. The next stage of this research will involve testing the direct influence of selected papers on the stability of diazotype records.

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