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# Suitability of silver birch bark as a natural source for cotton dyeing

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**Abstract**: Suitability of silver birch bark as a natural source for dyeing cotton. The study was carried out on dyeing cotton fabric with silver birch bark extract without a mordant and with the use of mordants - oxalic acid and inorganic salts of aluminum, tin, iron and copper. The color of the fabric was determined in the CIE L*a*b* system. Pale orange to salmon shades were obtained. In the case of iron and copper, a significantly different color was obtained, dark grayish and rusty, respectively. Color fastness tests were carried out using hot water, mineral acid, mild and hot washing, dry cleaning and natural exposure to sunlight. Excellent resistance to dry cleaning and mild and hot washing was found, as well as good resistance to mineral acid. The dyed fabric had the weakest, although still quite good, resistance to sunlight.

*Keywords*: silver birch bark, dye, mordant, color fastness

## INTRODUCTION

Birch (*Betula* L.) is a genus of trees and shrubs belonging to the birch family (Betulaceae). These plants usually grow in poor habitats in the northern hemisphere and are often pioneer species. They are cultivated as ornamental trees for their attractive bark and for utility purposes, as a source of valuable wood, e.g. for plywood (Hughes 2015), charcoal (Mencarelli et al. 2022) or papermaking (Zhao et al. 2019, Asikainen et al. 2010).

Birches have been used by people for many centuries. Their leaves essential oils have diuretic, antirheumatic and diaphoretic properties. They also improve metabolism and have a detoxifying effect on the circulatory system (Demirci et al. 2004, Kovač-Bešović et al 2009). Birch juice is the sap collected at the break of winter and spring directly from birch trees (Svanberg et al. 2012). One of the most original uses of birch was the use of whipping rods, hence the name birching for a type of corporal punishment (Zellick 1978).

Birch bark is one of the largest by-products of the wood and paper industry, the amount of which reaches up to 18% of the raw material (Rizhikovs et al. 2022). Most of the bark is landfilled as waste or used to generate heat through combustion (Zhao et al., 2020).
The chemical composition of bark is significantly different from wood tissue, while it contains more lignin, extractives and inorganics (Zhao et al. 2020). The most recognised and unique compounds of birch tar are betulin (Lup-20(29)-ene-3β,28-diol) and other triterenoids related to lupene or oleanane (Krasutsky 2006, Popov et al. 2016). Moreover, hydroxy and epoxy fatty acids can be found in birch bark suberin along with phenolic compounds (Zhao et al. 2020).

Historically, there were many known uses for birch bark, the most important of which was obtaining birch bark tar. This tar was used as a waterproofing agent, an adhesive, repairing or sealing resin, along with medical or even for chewing (Stacey et al. 2020). There are many

artefact found, like hafted stone or bone tools, ceramics or jewellery (Courel et al. 2018). Birch bark tar residues can be recognised basing on the detection of lupane-related triterpenoids and their thermal degradation products like betulin and dihydrobetulin derivatives (Courel et al. 2018, Perthuison et al. 2020). Due to the high content of lipophilic substances, birch bark burns easily even when not dried, hence its use as kindling (Stacey et al. 2020). Nowadays, numerous studies are being conducted on the use of various ingredients contained in birch bark. Suberic acids are suggested as an addition to adhesives for wood composites like particleboard (Tupciauskas et al. 2019, Makars et al. 2022, Rizhikovs et al. 2022) or plywood (Paze & Rizhikovs 2019). Extraction residue as an addition to particle boards (Jeżo & Wronka 2022). Birch bark extracts are tested for their antioxidative, anti-inflammatory and even probably anti-cancer properties (Pavlova et al. 2003, Alakurtti et al. 2006, Ou-Yang et al. 2023).

There are many recipes for dyeing fabrics using birch bark on the Internet, so they may have been known historically. Unfortunately, it is very difficult to find any scientific information on this subject, and this work is intended to at least partially fill this gap.

## MATERIALS

### Materials

Commercial pure cotton white fabric was used in the tests performed. The color and its uniformity were previously tested (Radomska & Radomski 2022) and determined as suitable for the experiments: $L^* = 93.10 \pm 0.08$, $a^* = -0.14 \pm 0.06$ and $b^* = -1.80 \pm 0.05$.

Silver birch (*Betula pendula* Roth) bark was obtained from the trunk of a 3-year-old tree cut down in a private garden in Rembertow district of Warsaw in January and seasoned for 6 months. Relative moisture content of the bark was determined as 3,0 % by oven drying at 105 °C. Bark extract was obtained by boiling 40 g of ground bark in 300 cm³ of freshwater. Bark was placed in a glass vessel, then in a water bath and kept at 98 °C for 90 min. Obtained extract was filtered with fast filter paper.

The research was conducted in a manner similar to that described in our previous paper (Radomska & Radomski 2022).

Mordants were prepared as 2 % solutions of following compounds:
Oxalic acid ($H_2C_2O_4 \cdot 2H_2O$), p.a.
Alum ($AlK(SO_4)_2 \cdot 12H_2O$), p.a.
Stannous chloride ($SnCl_2 \cdot 6H_2O$), p.a.
Copper(II) sulfate ($CuSO_4 \cdot 5H_2O$), p.a.
Ferric chloride ($FeCl_3 \cdot 6H_2O$), p.a.

Test reagent used for color fastness determination:
Perwol Renew liquid laundry detergent for synthetic & athletic clothes (Henkel),
Cyclohexane, p.a.
Hydrochloric acid (HCl, 36 %), p.a.

Potable freshwater from a private well (Rembertow district of Warsaw) was used in the experiments. Water properties were determined as pH 8.4, hardness 15.3 German degrees, alkalinity 7.2 German degrees.

### Mordanting

Raw fabric was cut into pieces (strips) measuring 210 × 40 mm. Five of the pieces were placed in 50 cm³ vials of PP and 40 cm³ of prepared mordant solutions was poured into the vials separately. The vials were placed in water bath and held for 30 min at 95 °C. Every 10 min all

vials were shaken by hand. After water bathing vials were left to cool down for 30 min, then each strip was pressed in order to remove the excess of used mordant solution without rinsing. An additional strip of cut fabric was left as a non-mordanted reference.

Dyeing

Each piece of fabric was placed in 50 cm³ vials of PP and 30 cm³ of birch bark extract was poured. The vials were placed it water bath and held for 60 min at 95 °C. Every 15 min all vials were shaken by hand. After water bathing vials were left to cool down for 30 min, then all strips were rinsed with freshwater.

Testing color fastness

After dyeing prepared strips of fabric were cut into seven pieces each measuring 30 × 40 mm. Color fastness tests were performed as follows:

**1. Hot water**

Five pieces of fabric treated with each mordant respectively and the untreated one were placed separately in PP 50 cm³ vials and 30 cm³ of tap water was poured. All vials were placed in water bath and held at 95 °C for 30 min. Every 10 min all vials were shaken by hand, then placed in cold water bath for 5 minutes to cool down. Each strip of fabric was rinsed using freshwater.

**2. Laundry in 50 °C *(mild wash)***

The liquid laundry detergent solution was prepared by dissolving 4 $cm^3$ of the detergent in 800 $cm^3$ of distilled water.

Five pieces of fabric treated with each mordant and the untreated one were placed separately in PP 50 cm³ vials and 20 cm³ of liquid laundry detergent solution was poured. All vials were placed in an oven and held at 50 °C for 70 min. Every 10 min all vials were shaken by hand, then placed in cold water bath for 5 minutes to cool down. Each strip of fabric was rinsed using freshwater.

**3. Laundry in 95 °C *(hot wash)***

The same liquid laundry detergent solution was used for mild and hot wash test.

Five pieces of fabric treated with each mordant and the untreated one were placed separately in PP 50 cm³ vials and 20 cm³ of liquid laundry detergent solution was poured. All vials were placed in water bath and held at 95 °C for 60 min. Every 15 min all vials were shaken by hand, then placed in cold water bath for 5 minutes to cool down. Each strip of fabric was rinsed using freshwater.

**4. Dry cleaning**

Five pieces of fabric treated with each mordant and the untreated one were placed separately in PP 50 cm³ vials and 10 cm³ of cyclohexane was poured. All vials were placed in water bath and held at 37 °C for 30 min. Each strip of fabric was placed in dryer for 5 min at 105 °C.

**5. Mineral acid**

The 0.5 % hydrochloric acid solution was prepared by diluting 40 ml of 5 % hydrochloric acid with 360 ml of distilled water.

Five pieces of fabric treated with each mordant and the untreated one were placed separately in PP 50 cm³ vials and 20 cm³ of hydrochloric acid was poured. All vials were placed in water bath and held at 98 °C for 30 min. Every 10 min all vials were shaken by hand, then

placed in cold water bath for 5 minutes to cool down. Each strip of fabric was rinsed using freshwater.

**6. UV exposure**

Five pieces of fabric treated with each mordant and the untreated one were placed on PVC board and clamped with clothes clip. The PCV board with fabric strips was exposed to natural sunlight outdoors for seven days. The weather during the research was:
Day 1: full sun
Day 2: full sun
Day 3: full sun
Day 4: overcast, moderate rain
Day 5: mostly sunny
Day 6: full sun
Day 7: mostly sunny

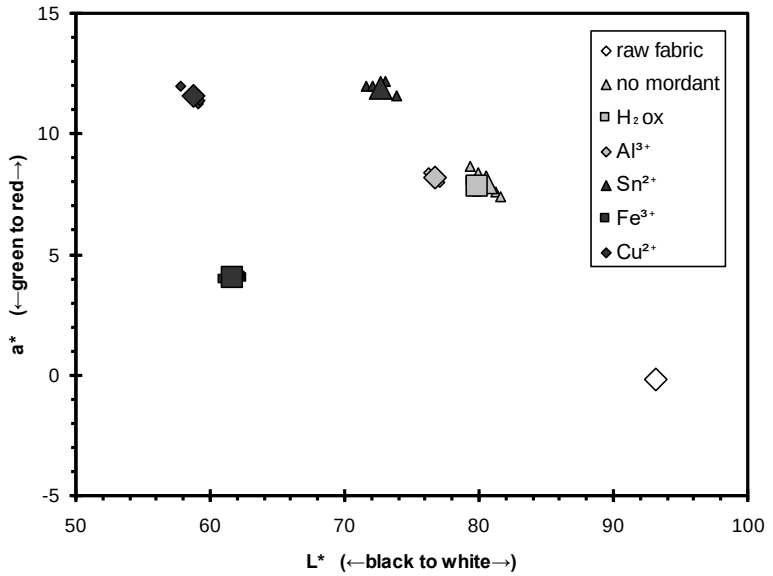
Color determination

Each sample was tested for color change after the process of dyeing and color fastness testing. The color determination was performed with the use of a Spectromaster Model 565-D spectrophotometer, in the CIEL*a*b* system, while the coordinates descirbe respectively: L* - brightness level, a* - hue on the green-red axis, b* - hue on the blue-yellow axis.

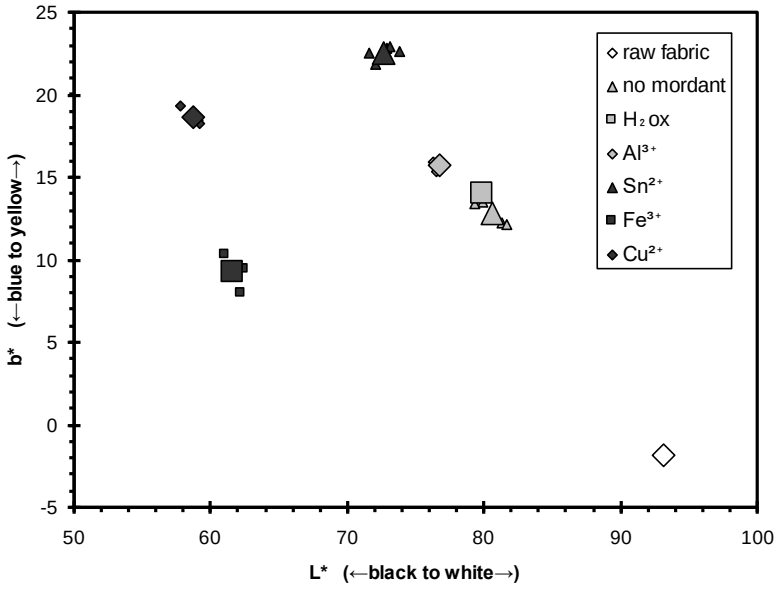
RESULTS

**Dyeing**

The results of dyeing are presented in the Fig. 1. For ease of readability, the data are presented in two coordinate systems of L*–a* and L*–b*. Smaller markers represent individual points and bigger ones represent mean values.




A




B

**Figure 1**. Color for dyed fabrics in L*-a* (A) and L*-b* (B) coordinate systems

No mordant: The color change was slight. Towards the red shade the change was approximately 7 units, in the direction of the yellow shade it was approximately 13 units. The brightness changed as the sample darkened to L=80.

Oxalic acid: In the case of oxalic acid used as a mordant, the results were almost identical as in the case of no mordant used. A slightly greater change in color in the direction of the yellow shade by approximately 13 units is the only noticeable difference.

Alum: In the case of alum used as a mordant, the results were also similar to the case with no mordant used. There was a change of the b* coordinate towards more intense yellow. The obtained shade was slightly darker.

Tin: In the case of tin based mordant the change of color was the most significant. The color change towards red shade occurred to a level of approximately 12 units, towards yellow it was over 20.

Copper: In the case of copper based mordant, the change in shade is also intense. The change of the a* coordinate towards red is comparable to the case of tin mordant. The change of the b* coordinate towards yellow is slightly smaller, giving less than 20 units. The brightness of the sample changed significantly. Samples treated with copper based mordant are the darkest among all obtained. The L parameter values were below 60.

Iron: In the case of iron based mordant the sample darkened noticeably, a little less than in the case of copper mordant, with the L parameter valued slightly over 60. The most characteristic feature of the samples treated with iron mordant is a very small color change towards both red and yellow. The change was the smallest among all tested samples, resulting in the grayish shade.

## Color fastness

The color fastness tests were carried out in series for each type of mordant used, along with the reference series. The results are showed in the following figures, separately for each mordant.

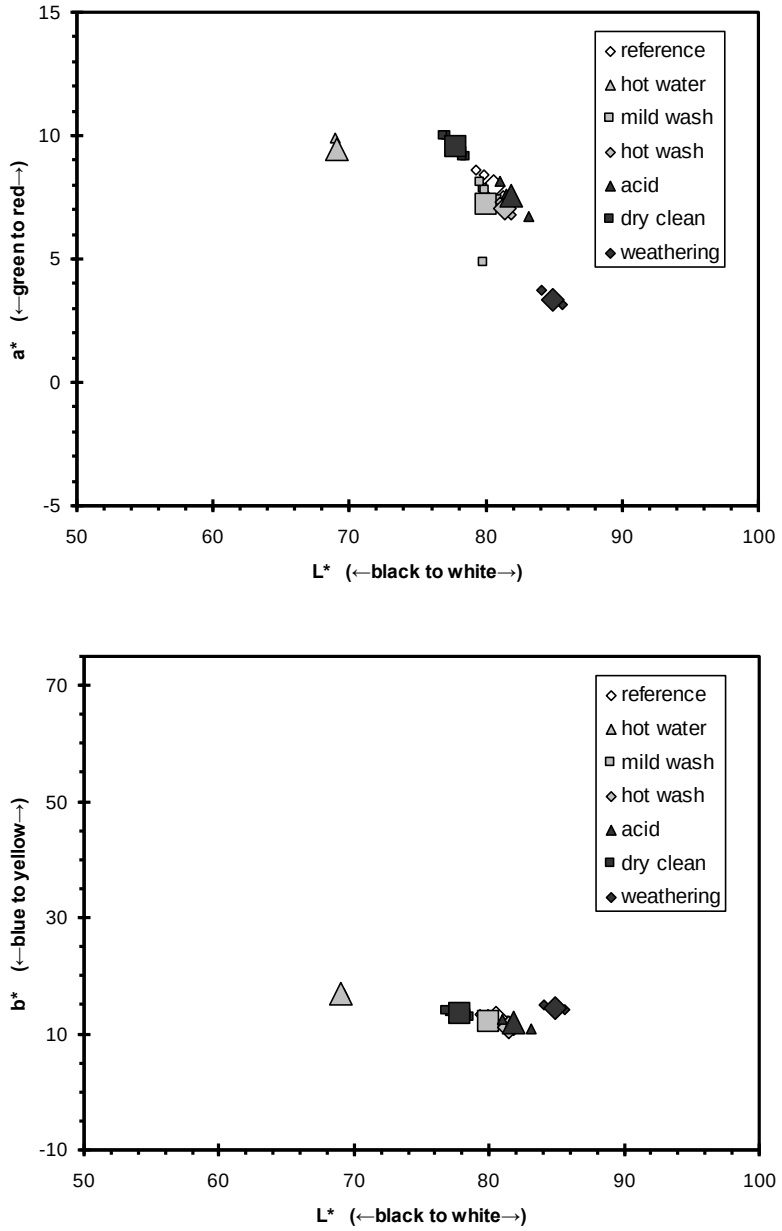
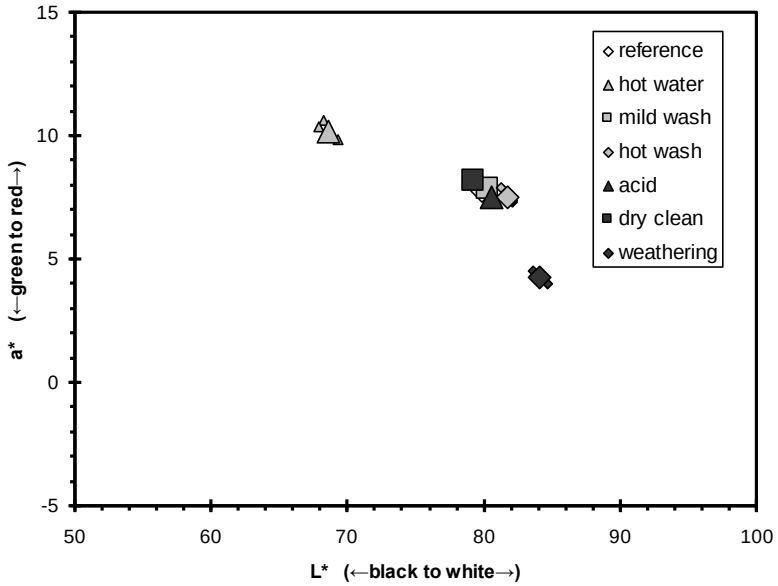
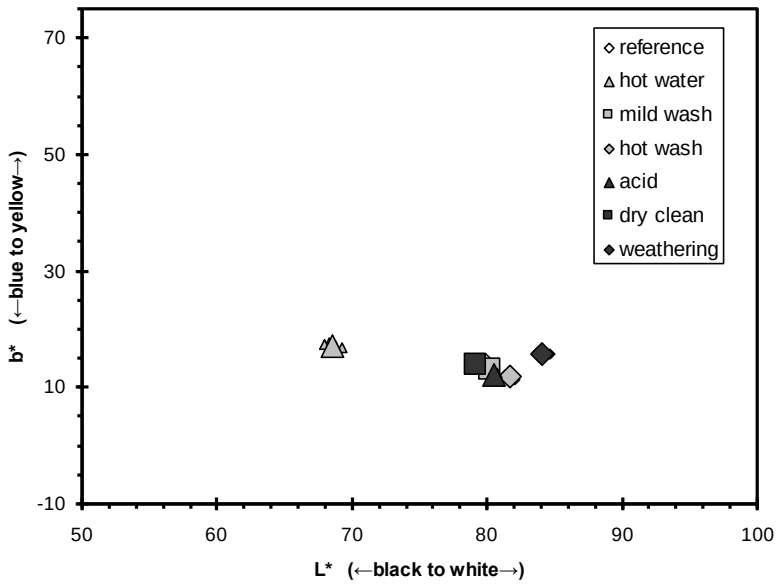



**Figure 2.** Color fastness for untreated fabrics in L*-a* (A) and L*-b* (B) coordinate systems

No mordant (Fig.2): In most cases the color is well durable. The biggest change in compare with the initial samples was observed in the case of weathering, where brightening and decrease in red shade intensity were obtained. Hot water caused darkening with increase in both a* and b* coordinates. In the case of dry cleaning the sample slightly darkened and the a* coordinate increased towards red shade.



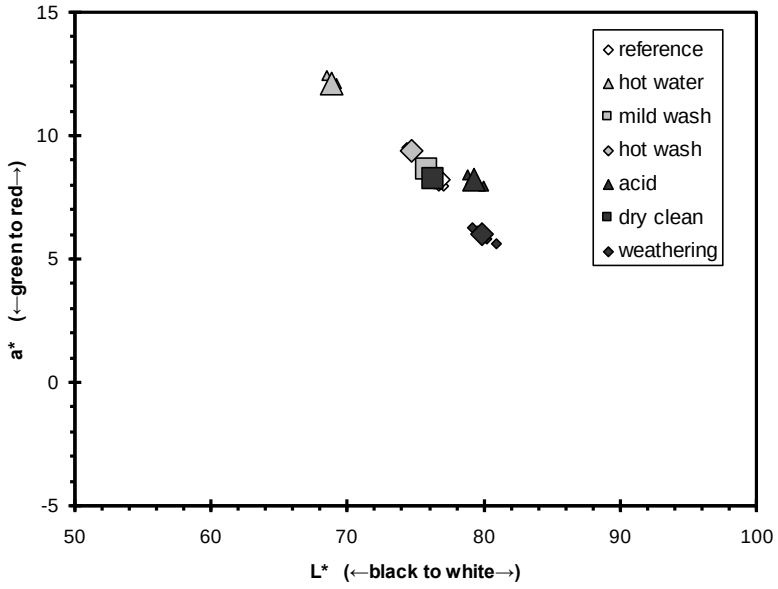

A



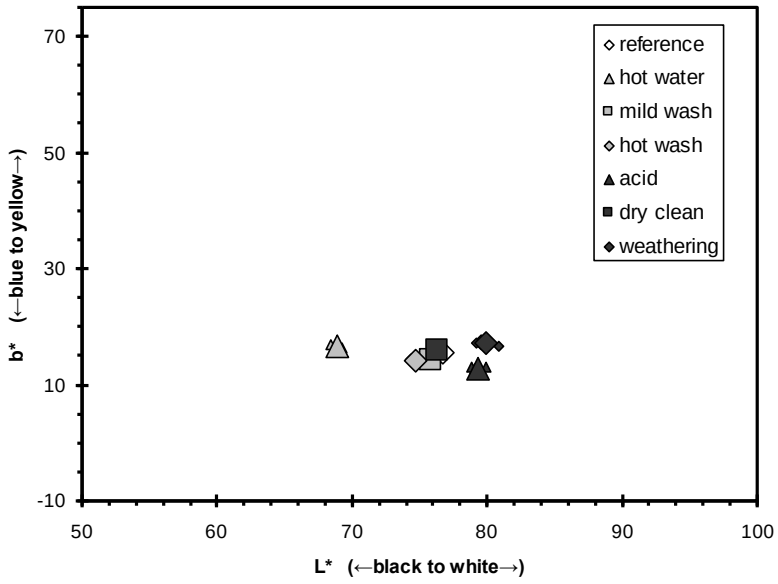

B

**Figure 3.** Color fastness for oxalic acid treated fabrics in L*-a* (A) and L*-b* (B) coordinate systems

Oxalic acid (Fig.3): The initial color of the samples after dyeing did not differ much from the samples with no mordant used. The durability of the color is satisfactory. The significant change in color in compare with the initial samples occurred only in case of weathering, where a slight brightening and a decrease of red shade were observed, and hot water, where a strong darkening with obtaining slightly more intense both red and yellow colors was observed.




A




B

**Figure 4.** Color fastness for alum treated fabrics in L*-a* (A) and L*-b* (B) coordinate systems

Alum (Fig.4): The color durability can be described as medium. The brightening of the color was observed in the case of acid and weathering. There was almost no change in shade for acid, while for weathering the a* coordinate decreases and the b* coordinate increases slightly. In the case of mild wash, hot wash and dry cleaning the changes are minimal, hard to notice for average user. The most significant change was caused by hot water, the samples became noticeably darker while the hue changed towards more intense red.

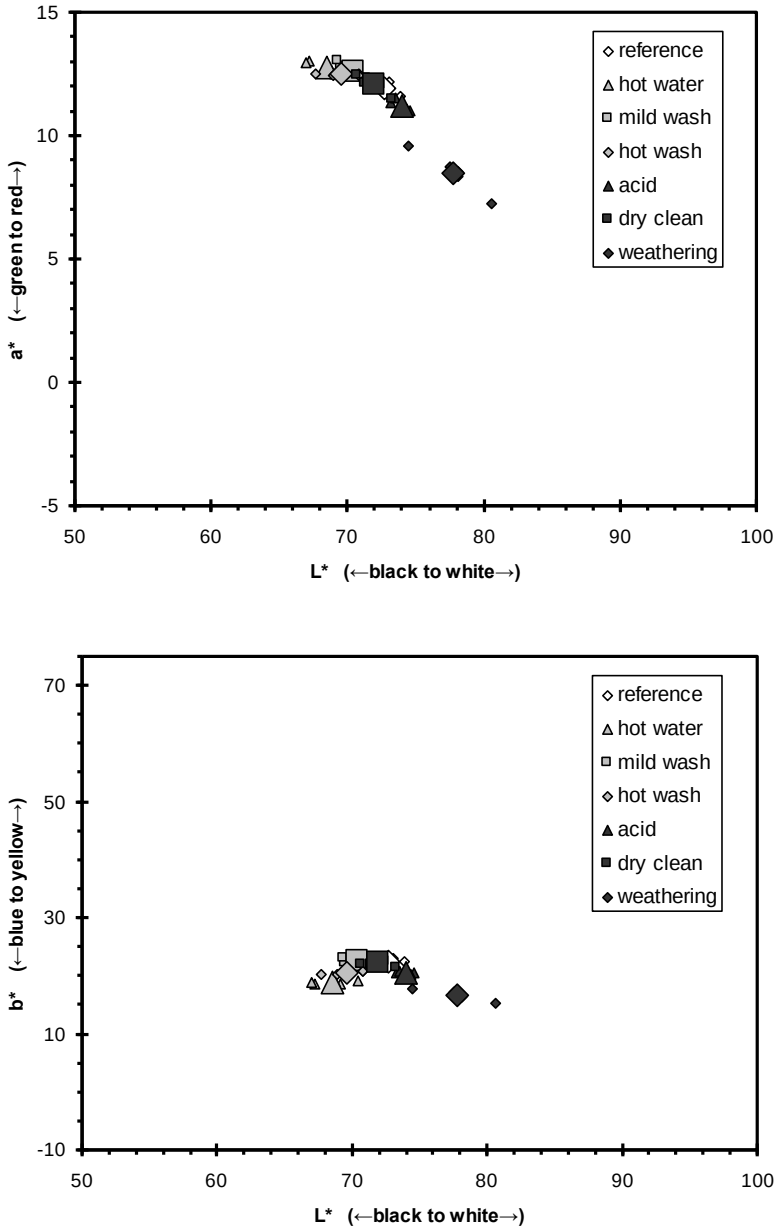
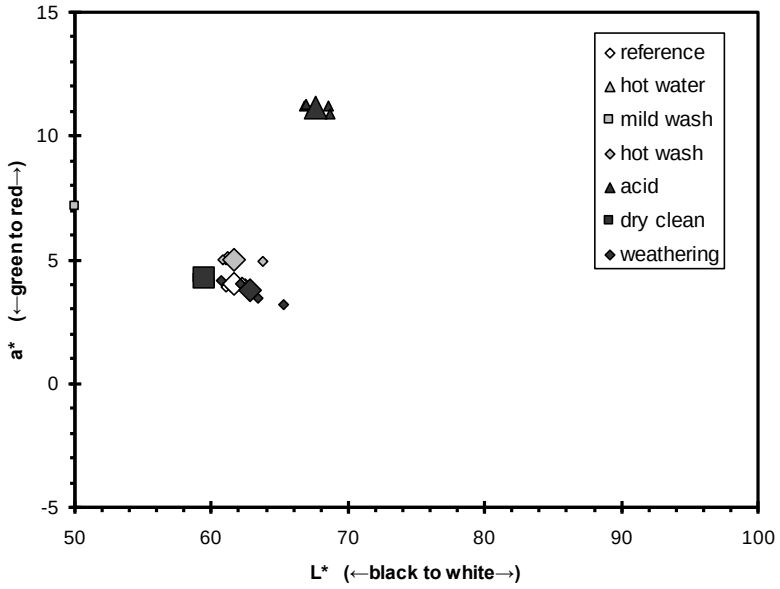
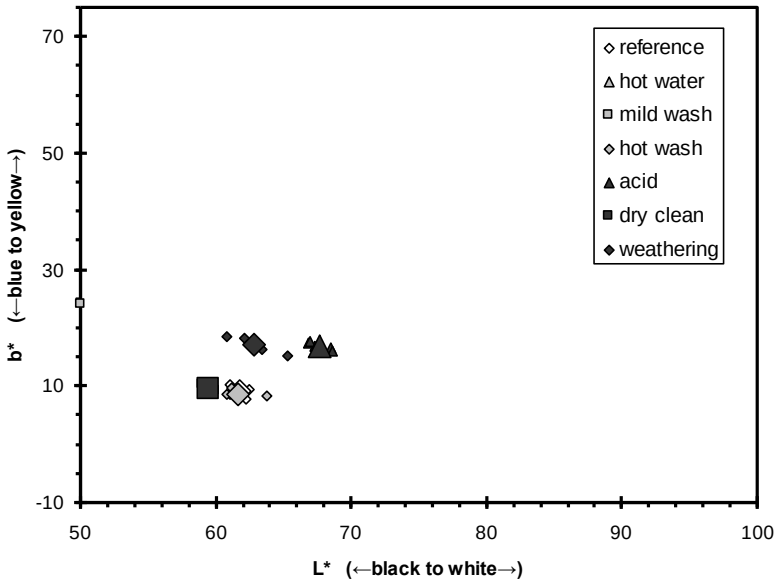



**Figure 5.** Color fastness for tin chloride treated fabrics in L*-a* (A) and L*-b* (B) coordinate systems

Tin (Fig.5): The color turned out to be well durable. The sample was the lest resistant to weathering, which resulted both in loss of color, mainly red shade, and brightening in compare to the original sample. The yellow shade remained almost the same. Other used methods caused little to no change in color. The shade does not change significantly, while the brightness of the samples increases slightly in the case of acid. Hot water, mild wash, hot wash and dry cleaning caused a slight decrease in brightness, which was most significant in the case of hot water.



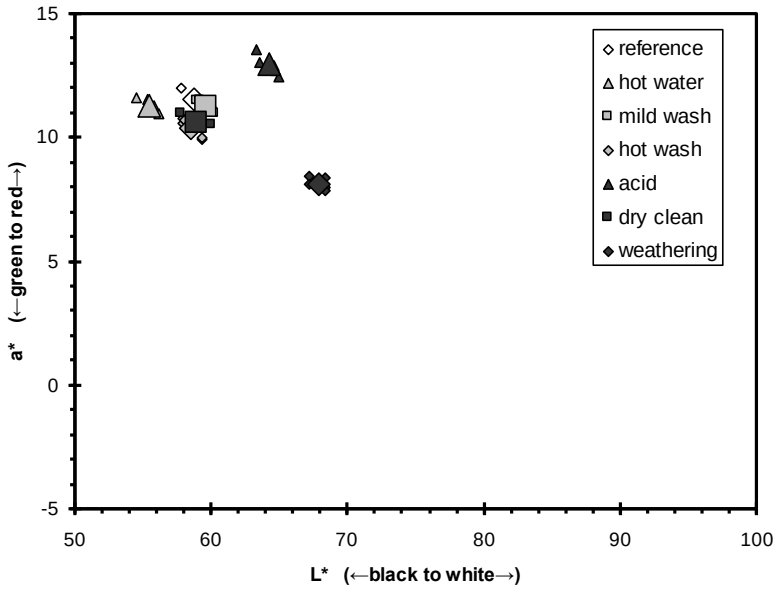

A



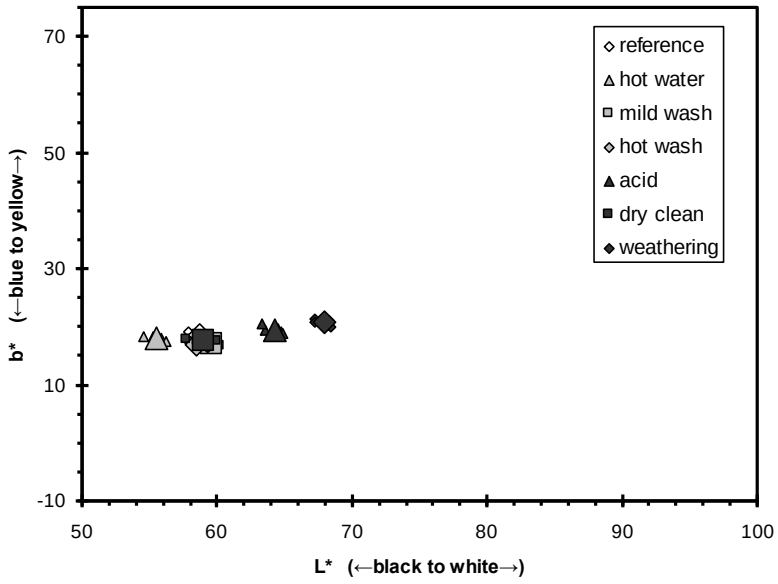

B

**Figure 6.** Color fastness for ferric chloride treated fabrics in L*-a* (A) and L*-b* (B) coordinate systems

Iron (Fig.6): The fastness of the color obtained with iron based mordant is very good. Most of the used tests did not cause a significant change in color. Minimal changes in shade were noticeable in the case of weathering. Dry cleaning resulted in minimal darkening. The biggest change occurred in the case of acid, which caused the sample to take on a noticeably redder shade. The b* coordinate changed slightly, there was a little change towards yellow shade in the case of weathering and acid. In the case of acid sample was also brightened.




A




B

**Figure 7.** Color fastness copper sulfate treated fabrics in L*-a* (A) and L*-b* (B) coordinate systems

Copper (Fig.7): Before the aging process the samples were very dark. Hot water, hot wash, mild was and dry cleaning did not cause any significant change in color. Similar to other cases, acid caused brightening and intensifying the red shade. In the case of weathering the brightening was the strongest, there was a minimal increase in the b* coordinate and noticeable decrease in the a* coordinate.

## CONCLUSIONS

The compound in the bark of silver birch are suitable for cotton dyeing. Mordants tested showed various efficiency, as the colors obtained with oxalic acid or alum were not clearly different from that of non mordanted cotton fabric, giving similar, pale-orange or salmon like shades. Much more intense color, although still very light was obtained using tin based mordant. Copper and iron based mordants may be recognized as color modifiers, as the difference in color obtained was much more pronounced. Copper based mordant gives much darker and deeper, rusty shade while iron based give unattractive dark grayish colors. Silver birch bark gives definitely less shades than apple tree bark, investigated in our previous paper (Radomska & Radomski 2022).

On the other hand, the obtained colors were noticeably more durable than those obtained in case of dyeing fabric with apple tree bark. In the case of mild or hot washing, or dry cleaning, very good color fastness was found, independently of the mordant used. Hot water without detergents did not cause discoloration in most cases, but in the case of non mordanted fabric and mordanted with oxalic acid or alum surprisingly pronounced darkening was observed. UV radiation was found to be the most color degrading factor, except the case of iron based mordant exposure, which was the only one case of color resistant to UV exposure. In this case the most color changing factor was mineral acid, causing fabric lightening, while the shade intensity was found increasing. The only other case of mineral acid pronounced action was the fabric mordanted with copper sulfate, while much slighter than UV radiation.

Taking into consideration much larger volume of birch timber in wood industry and satisfactory of obtained color fastness, use of silver birch bark to eco-dyeing of fabric seems to be a very attractive perspective of application. Moreover, water extraction of the bark does not exclude obtaining further compounds, such as suberin or triterpenes, which makes it possible to develop biorefinery technology using birch bark as a raw material.

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**Streszczenie**: *Przydatność kory brzozy brodawkowatej jako naturalnego surowca do barwienia bawełny*. Przeprowadzono badanie barwienia tkaniny bawełnianej ekstraktem z kory brzozy brodawkowatej bez zaprawy oraz z zastosowaniem zapraw – kwasu szczawiowego oraz soli nieorganicznych glinu, cyny, żelaza i miedzi. Barwa tkaniny oznaczana była w systemie CIE L*a*b*. Uzyskano odcienie blado pomarańczowe do łososiowych. W przypadku żelaza i miedzi uzyskano zdecydowanie odmienną barwę, ciemny szary i rdzawy odpowiednio. Przeprowadzono testy trwałości barwy stosując gorącą wodę, kwas mineralny, łagodne i gorące pranie, czyszczenie na sucho oraz naturalną ekspozycję na światło słoneczne. Stwierdzono znakomitą odporność na czyszczenie na sucho oraz łagodne i gorące pranie oraz dobrą na działanie kwasu mineralnego. Najsłabszą, choć wciąż dość dobrą odporność barwiona tkanina wykazywała na działanie światła słonecznego.

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