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Scouring Test of Sheep Wool Intended for Sorption

Abstract
Sheep wool intended for sorption examination was scoured using three procedures consisting of (1) an ultrasonic tempered bath with tap water, (2) the same as (1) but with detergent, and (3) dichloromethane extraction. The loss of wool mass and removal of Cu, Zn and Pb-cations by the scoured wool as a sorbent were tested. While the loss of mass indicated consistency among all procedures, the cation removal was slightly variable. However, the differences are acceptable, concluding that an ultrasonic water bath without any agent is most favoured. The omission of a detergent or carcinogenic solvent can make the scouring process cleaner and environmentally-friendlier.

Key words: *sheep wool, scouring, sorption, testing.*

Introduction

Several methods aim at the recuperation of technological wastes or elimination of environment contaminants. One of them is the application of various adsorbents by the separation of required or undesirable constituents from mixtures. A whole branch of science deals with research of the preparation and efficiency of sorbents, and it is the topic of an increasing number of studies [1]. Sorbent cost derived from material expenses, technological demands and sorptive efficacy are the determining factors of potential applications. Efforts to reduce costs are reflected in the examination of natural material exploitation, especially when applied to technological by-products or even wastes. The works published focus on the investigation of lignocellulosic biosorbents [2] as well as biopolymers of animal origin, especially of the keratin type [3]. The effort to improve the sorption properties of biopolymers involves chemical or physical modifications with aim to introduce new active groups into their structure. After being properly modified, sheep wool unusable in the textile industry could be applied as a sorbent as well. However, each modifying procedure requires the wool to be scoured.

The scouring of raw wool fibres is a strenuous task since it contains considerable amounts of impurities. While the authors of studies on wool describe the key modifying procedures used in detail, they refer only briefly – if at all – to the concept of wool scouring before experiments. In the papers previously published, the most frequent methods are degreasing by Soxhlet extraction using organic solvents [4, 5]. Also the application of non-ionic detergent is reported [6].

The results from a few of the works published show an obvious improvement of the scouring effect by the application of ultrasound. Although the principle of the ultrasound cleaning effect has been known for many years, the use of ultrasonic energy for textile washing has not been commercially developed [7]. One advantage of ultrasonic scouring is the avoidance of wool aggressive movement in the cleaning bath provoking the entanglement and felting of wool fibres with hand washing. A possible improvement of ultrasonic scouring using a detergent and higher temperature of the ultrasonic bath was examined by Hurren [8]. He found that both temperature and detergent concentration in the bath can be optimised to maintain cleaning efficiency at an adequate level. The detergent improves the wetting of wool fibres with contaminants and enhances the ultrasonic effect. Ultrasound also increased the rate of dye uptake by the fibres without a significant adverse impact on the fibre mechanical properties. Since wool dyeing is the most frequent issue in the textile industry, there are several papers dealing with this topic in scientific literature e.g. [9].

The results of Hurren [8] and Kadam et al. [10] indicated that a lower power of the ultrasound water bath had no impact on the fibre structure, and at a higher level some transverse cracks were observable on the cuticle. Similarly when distilled water was used with a detergent at low ultrasound power, no visible change in the fibre surface was observed. However, a high power level caused transverse and longitudinal cracks over the fibre surface. Peila et al. [11] examined the ultrasonic effect on washing quality for cotton and wool fabrics using a mixed detergent

and temperature of the ultrasonic bath of 25 °C and 60 °C. They found that temperature is a parameter with a high influence on ultrasonic cavitation. Higher temperatures restrain the cavitation, and better results were achieved at a lower temperature and shorter time of 10 min. As observed by Li et al. [12, 13], under certain conditions the application of ultrasound can even improve some wool properties. Compared to classical mechanical washing, ultrasound decreases fibre entanglement due to the reduction of fibre migration in the washing bath [14]. Bahtiyari et al. [15] scoured wool fleece consecutively in five washing steps with and without ultrasound. Comparable results were observed after the third washing step using ultrasound and with the fifth step with no ultrasound. Examination of a bath temperature of 50 °C with ultrasound for 30 min showed as an alternative for non-ultrasonic consecutive scouring. Interesting results were also observed for washing at 35 °C with only water and applying ultrasound. A detailed study on the optimal parameters of ultrasonic scouring using soap and soda is described by Czaplicki and Ruszkowski [16]. Demonstrating on alpaca wool, the work pointed out the benefits of such a procedure, consisting, besides others, especially in the avoidance of entanglement. Kunik et al. [17] combined high-energy discrete processing (HDP), the essence of which is the phenomenon of electric discharge cavitation, with a mixture of detergents and sulfoxide, obtaining a good result.

None of the papers published was devoted to scouring for sorption intention. Currently this aspect has become topical since sheep wool after being irradiated by an accelerated electron beam showed

interesting sorption properties towards some substances [18, 19].

In the present study, the main purpose was to optimise the scouring of sheep wool before being irradiated and then examined for the sorption/removal of a metal cation from the solution. Since several chemicals with a potential impact on the sorption characteristics of scoured wool, hitherto untried, are used in the industrial procedure, industrial scouring was not taken into consideration as a reference. In addition, scouring using real industrial facility runs with a large wool amount excludes the identification of the origin, breed or age of individual sheep. Therefore even if fibres of the same thickness were selected, they could hardly present material suitable for comparison with an actual laboratory sample. Furthermore an effort to reduce the usage of any chemicals in laboratory scouring could be a suitable contribution to the protection of the environment as well. Therefore the work included scouring in a tap water ultrasonic bath with or without a commercial detergent recommended for wool textile, as well as Soxhlet extraction with dichloromethane, where the primary parameter tested was wool mass loss during the scouring, and the second crucial parameter was the sorption of selected heavy metal cations by the cleaned wool.

Materials and methods

Experiments were performed with the wool of the Tsigai (meat – dairy breed) and Suffolk (meat breed) crossbreed. The region of fleece sampling from the animal body could not be exactly defined however, and fibres were randomly taken from several parts of the fleece to obtain a representative sample as far as possible. First the sheared wool taken and mixed was manually pre-cleaned from coarse dirt (excrements, dirt, food, etc.) and then rinsed several times in tepid water until it became visually clear. Before the scouring process, the pre-cleaned wool was stored in a dark, dry and airy place. The thickness of the fibre was in the range of 25-33 µm.

All reagents used in the study were of analytical grade purity. Dichloromethane, copper (II) sulfate pentahydrate $CuSO_4 \cdot 5 H_2O$, zinc (II) sulfate heptahydrate $ZnSO_4 \cdot 7 H_2O$ and lead (II) nitrate $Pb(NO_3)_2$ were supplied by Centralchem, Slovakia. Test solutions of those salts to

be applied for the sorption experiments were prepared by diluting the corresponding stock solutions with distilled water to required concentrations.

Standard stock solutions of Cu(II), Zn(II) and Pb(II) for AAS spectrometry, each with a concentration of 1000 mg·dm⁻³ in 3 % HNO_3, were supplied by Agilent Technologies, USA. The stock solutions were diluted to required concentrations with distilled water. Nitric acid (67%) was supplied by Analytika, Czech Republic.

The laundry agent Perwoll (Sport & Active) was bought in a retail outlet (Billa, Nitra), with the following declared composition: 5-15% anionic detergents, < 5% non-ionic detergents, soap, phosphonates, enzymes, perfume, and preserving agents.

Scouring samples

Three different washing methods were applied as follows: (1) ultrasonic washing in tap water, (2) ultrasonic washing in tap water with detergent, and (3) extraction with dichloromethane in Soxhlet apparatus. Each washing experiment was repeated 5 times to minimise the influence of various fibre thicknesses. An amount of 3 g of wool was taken for all parallel experiments.

(1) Ultrasonic washing in tap water: A weighed amount of about 3 g of wool was placed in a netted bag and immersed in tap water heated to 40 °C in an ultrasonic bath of 5 dm³ (Kraintek, K5LE, Slovak Republic, power of 350 VA, output of 450 W, frequency of 38 kHz, heating from 20 to 80 °C, timer from 0 to 90 min). A maximal adjustable intensity level of 9 was used. After 10 min washing, the water was changed and the same washing cycle repeated. Then the bag with wool was removed and the sample rinsed with 5 dm³ of distilled water in a vessel. After the washing, the wool was spread on filter paper and free-dried at room temperature and humidity for 24 hours.

(2) Ultrasonic washing in tap water with the detergent was performed under the same conditions as the procedure described above for (1). The dosage of the detergent followed the manufacturer's recommendation, i.e.: 40 cm³ of Perwoll per 5 dm³ of water. After each 10-min washing cycle, the

sample was rinsed in running water to remove the detergent. After the completion of washing, the sample was washed with 5 dm³ of distilled water, spread on filter paper and free-dried at room temperature and humidity for 24 hours.

(3) Soxhlet extraction was carried out with a 3 g wool sample put in a paper thimble. A volume of 250 cm³ of dichloromethane was used, and the extraction ran for 4 hours (14 drain-off cycles). Then the wool sample was removed from the thimble, and the solvent could evaporate freely in a fume hood. Subsequently the sample was washed twice with 5 dm³ of distilled water, spread on filter paper, and dried freely at room temperature and humidity for 24 hours.

After each scouring procedure, the samples were finally dried in an oven (Binder, ED 115, Germany) at 40 °C for 24 hours, and when the temperature dropped to room temperature, the samples were weighed. Before further sorption experiments, the wool samples were stored in zip PE pockets to prevent moisture adsorption from air.

The percentage of the decrease in wool mass (Δ mass) after the scouring was taken as the criterion of scouring efficiency, calculated using the following *Equation (1)*:

$$\Delta mass = \frac{m_0 - m_1}{m_0} \cdot 100\% \qquad (1)$$

Where, m_0 is the mass of the sample taken for scouring in g, and m_1 – the mass of the sample after scouring in g.

Sorption experiments

Sorption experiments were carried out with test solutions of Cu^{2+}, Zn^{2+} and Pb^{2+} individually with the following concentrations: $CuSO_4 \cdot 5H_2O$ solutions containing (12.7-50.8) mg Cu/dm³, $ZnSO_4 \cdot 7 H_2O$ solutions containing (6.54-26.16) mg Zn/dm³, and $Pb(NO_3)_2$ solutions containing (20.7-103.5) mg Pb/dm³. Dry wool fibres (0.2 g) were cut to 3-5 mm and placed in a small 27 cm³ glass cup with a cap. A volume of 12 cm³ of the test solution was added to the wool sample. To ensure a thorough soaking of the wool sample, the fibres were immersed in the solution using a glass rod. The sample was kept in contact with the test solution for 24 hours and shaken for the first 6 hours on a laboratory horizon-

Table 1. *Survey of experimental conditions under AAS measurement.*

Determined element	Cu	Zn	Pb
Wavelength emitted by lamp, nm	324.8	213.9	217.0
Flow acetylene/air, dm³/min	2.0/13.5	2.0/13.5	2.0/13.5
Concentration of calibration solutions, mg/dm³	2; 4; 8; 10	0.3; 0.5; 1; 1.5	1; 5; 10; 20
Concentration of calibration solutions used to optimise signal for absorbance of 0.2, mg/dm³	1.5	0.3	5

Table 2. *Loss of wool mass under scouring.*

No.	Scouring procedure	Average decrease in mass, %	Standard deviation, % for α = 0.05	Relative deviation, %
1	Ultrasonic water bath	5.53	0.7136	12.9
2	Ultrasonic water bath with Perwoll	11.47	0.8598	7.50
3	Dichloromethane extraction	9.67	1.1478	11.79

Table 3. *Test consistency of loss in mass under sheep wool scouring for α = 0.05 level; n = 5; $t_{crit. n = 5}$ = 2.776.*

No.	Pair compared for scouring procedure effect	t-criterion calculated	Statement
1	Ultrasonic water bath	t_{1-2} = 0.2617	Consistency
2	Ultrasonic water bath with Perwoll		
1	Ultrasonic water bath	t_{1-3} = 0.6425	Consistency
3	Dichloromethane extraction		
2	Ultrasonic water bath with Perwoll	t_{2-3} = 0.4016	Consistency
3	Dichloromethane extraction		

Table 4. *Efficiency of Cu(II) removal for scoured sheep wool.*

No.	Scouring medium	Percentage of Cu(II) removal from bath for initial Cu(II) concentration, mg·dm⁻³						
		12.7	19.05	25.4	31.75	38.1	44.45	50.8
1	Ultrasonic water bath	95.66	96.11	94.71	93.28	89.65	83.59	83.89
2	Ultrasonic water bath with Perwoll	98.02	98.41	98.19	95.51	92.26	93.72	85.51
3	Dichloromethane extraction	98.30	98.98	99.09	97.11	96.87	98.08	93.51

tal shaker (TE, Kavalier, Czech Republic) at room temperature. After 24 h, the remaining solution was filtered through KA5 filter paper and used for analysis of the residual amount of the metal cation. Every sorption experiment was repeated 5 times in order to reduce the standard deviation.

Determination of residual concentration of metal cation

The test solution concentration in the filtrate was determined by atomic absorption spectrometry (AAS) using a spectrometer – 240 FS AAS (Agilent Technologies, USA). Between measurements the spectrometer was washed with 0.1% HNO_3. The device operative parameters under measurement are given in *Table 1*.

The percentage of ion removal of the scoured sheep wool was calculated from the concentrations measured as the removal efficiency (RE) using the following *Equation (2)*:

$$\Re = \frac{x_0 - x_1}{x_0} \cdot 100\% \qquad (2)$$

Where, x_0 is the concentration of relevant added ion in the solution in mg/dm³, and x_1 is the residual concentration of relevant ion in the solution after contact with the wool sample in mg/dm³.

Every sorption experiment was repeated 5 times.

Testing of consistency

Student's *t*-criterion was used to test the consistency of results obtained and calculated using the following *Equation (3)* [38]:

$$t_{A-B} = \frac{|x_A - x_B| \cdot \sqrt{n-1}}{\sqrt{s_A^2 + s_B^2}} \qquad (3)$$

Where, t_{A-B} is the *t*-criterion for statistical sets A and B under comparison, x_A-x_B –

the difference in arithmetic averages for statistical sets A and B, *n* is the number of repetitions, and s_A, s_B are the standard deviations from the arithmetic averages for statistical sets A and B estimated according to Dean-Dixon [20].

Results and discussion

Testing of scouring procedures
Depending on the breed, age and season, sheep wool contains up to 30 % of grease. The wool lipid components are intimately associated with the surface proteins [18] and make the wool hydrophobic. In some processes, such as the sorption of polar substances from the solution, the hydrophobicity obstructs the necessary contact of the sorbent with the sorbate. It is presumable that micro-streaming coming from the cavitation bubble oscillation and following the micro-jetting could erode the surface lipid layer to such a degree that the necessary contact of the polar sorbate with the wool is attained.

An ultrasonic bath temperature of 40 °C was selected as it is close to the melting temperature of lanoline – 39.7 °C, as measured by Hanzlíková et al. [21], and to the boiling temperature of dichloromethane (39.6 °C). Together with the observation of Peila et al. [11] that higher temperature restricts cavitation and that a time of 10 min showed better results than a longer one, we considered 40 °C temperature and a time of 10 min per 1 cycle as probably optimum. The wool mass reduction for individual scouring procedures was considered as the indicator for cleaning efficacy. Related data are summarised in *Table 2*.

The differences between the scouring effects (*Table 2*) appear relatively large, and the combination of ultrasound with detergent produced the best result. It indicates the support of ultrasound for deeper penetration of the detergent into the wool fibre structure with following solubilisation of lipids in comparison with dichlomethane. From a subjective view, these results should not be identical. Therefore we examined this hypothesis by testing the data using Student's t-criterion to obtain an objective review. The t-rate estimated was compared to the critical t-value for n = 5. The results obtained are showed in *Table 3*.

Since the t-criteria do not exceed the critical value of 2.776 in any of the cases, we have to acknowledge that the procedures used are not significantly different and provide consistent results.

Testing of sorption

For the intended application, the sorption of the test substances was a coessential criterion. Therefore we examined how the sorption of some cations manifests in the wool scoured by the procedures applied. The range of the test cation concentrations was designed so that the residual concentration falls within the linear section of the relevant AAS calibration curve. From these data, the removal efficiency was calculated. Results for Cu(II) sorption removal are displayed in *Table 4*. The data in *Table 4* underwent testing for consistency, the related results of which are given in *Table 5*.

The statements in *Table 5* show that the pair consisting of an ultrasonic water bath and ultrasonic water bath containing Perwoll (t_{1-2}) gives an indecisive result overall due to the numbers for "consistency" and "inconsistency" being the same and one case being close to inconsistency. On the other hand, the pair involving ultrasonic water and dichloromethane extraction (t_{1-3}) show "inconsistency" for all concentrations applied. The consistency tested for both the ultrasonic water bath containing Perwoll and dichloromethane (t_{2-3}) gives the prevalence of consistency over inconsistency (3:2), with two cases close to inconsistency. Thus an unambiguously different effect can be declared only between the ultrasonic water bath and dichloromethane extraction (t_{1-3}).

The sorption effect of the wool samples on Zn(II) removal is summarised in *Table 6*. *Table 7* shows the tested hypothesis on consistency for relevant Zn(II) removal.

Unlike Cu-sorption by differently scoured wool, the consistency of the scouring procedures under Zn(II) sorption efficiency does not show prevalence in any case. The ultrasonic water bath and ultrasonic water bath containing Perwoll (t_{1-2}) pair is the closest to some kind of balance of consistency to inconsistency, showing a ratio of 3:4. The ultrasonic water bath and dichloromethane extraction (t_{1-3}) pair similarly gives a close prevalence of inconsistency over consistency, while the pair Perwoll ultrasonic water and dichloromethane extraction (t_{2-3}) provides inconsistency for each concentration.

The highest concentrations within AAS analyses could be applied for Pb(II). Data on Pb sorption by the wool samples are

Table 5. Results of consistency testing for Cu(II) removal efficiency for sheep wool scoured in an ultrasonic water bath (1), ultrasonic water bath containing Perwoll (2) and by dichloromethane Soxhlet extraction (3).

Initial concentration of Cu(II), mg·dm⁻³	Pair	t-criterion calculated, $t_{crit\ for\ n=5}$ = 2.776	Statement
12.7	t_{1-2}	5.339	Inconsistency
	t_{1-3}	6.664	Inconsistency
	t_{2-3}	0.910	Consistency
19.05	t_{1-2}	2.604	Close to inconsistency
	t_{1-3}	3.270	Inconsistency
	t_{2-3}	4.058	Inconsistency
25.4	t_{1-2}	7.587	Inconsistency
	t_{1-3}	14.346	Inconsistency
	t_{2-3}	2.605	Close to inconsistency
31.75	t_{1-2}	2.5183	Consistency
	t_{1-3}	4.296	Inconsistency
	t_{2-3}	2.741	Close to inconsistency
38.1	t_{1-2}	1.058	Consistency
	t_{1-3}	12.492	Inconsistency
	t_{2-3}	1.833	Consistency
44.45	t_{1-2}	3.515	Inconsistency
	t_{1-3}	5.028	Inconsistency
	t_{2-3}	5.465	Inconsistency
50.8	t_{1-2}	0.3734	Consistency
	t_{1-3}	3.802	Inconsistency
	t_{2-3}	1.987	Consistency

Table 6. Efficiency of Zn(II) removal by scoured sheep wool.

No.	Scouring medium	Percentage of Zn(II) removal from bath for initial Zn(II) concentration, mg·dm⁻³						
		6.54	9.81	13.08	16.35	19.62	22.89	26.16
1	Ultrasonic water bath	95.97	96.13	94.72	93.99	89.87	90.76	85.23
2	Ultrasonic water bath with Perwoll	95.46	95.31	88.35	93.69	85.12	86.73	83.18
3	Dichloromethane extraction	96.71	96.86	93.14	96.87	93.62	93.11	91.98

Table 7. Results of consistency testing for Zn(II) removal efficiency of sheep wool scoured in an ultrasonic water bath (1), an ultrasonic water bath containig Perwoll (2) and by dichloromethane Soxhlet extraction (3).

Initial concentration of Zn(II), mg·dm⁻³	Pair	t-coefficient calculated, $t_{crit\ for\ n=5}$ = 2.776	Statement
6.54	t_{1-2}	1.694	Consistency
	t_{1-3}	2.699	Close to inconsistency
	t_{2-3}	4.401	Inconsistency
9.81	t_{1-2}	1.505	Consistency
	t_{1-3}	1.548	Consistency
	t_{2-3}	3.066	Inconsistency
13.08	t_{1-2}	3.989	Inconsistency
	t_{1-3}	1.879	Consistency
	t_{2-3}	2.822	Inconsistency
16.35	t_{1-2}	0.442	Consistency
	t_{1-3}	5.142	Inconsistency
	t_{2-3}	6.667	Inconsistency
19.62	t_{1-2}	6.805	Inconsistency
	t_{1-3}	3.386	Inconsistency
	t_{2-3}	9.421	Inconsistency
22.89	t_{1-2}	6.805	Inconsistency
	t_{1-3}	4.025	Inconsistency
	t_{2-3}	12.234	Inconsistency
26.16	t_{1-2}	3.624	Inconsistency
	t_{1-3}	4.677	Inconsistency
	t_{2-3}	6.497	Inconsistency

Table 8. Efficiency of Pb(II) removal from scoured sheep wool.

No.	Scouring medium	Percentage of Pb(II) removal from bath for initial Pb(II) concentration, $mg \cdot dm^{-3}$				
		20.7	41.4	62.1	82.8	103.5
1	Ultrasonic water bath	99.7	99.16	98.59	88.39	77.81
2	Ultrasonic water bath with Perwoll	99.59	99.45	98.48	91.35	81.93
3	Dichloromethane extraction	99.01	99.07	99.17	97.52	97.37

Table 9. Results of consistency testing for Pb removal efficiency of sheep wool scoured in an ultrasonic water bath (1), an ultrasonic water bath containig Perwoll (2) and by dichloromethane Soxhlet extraction (3).

Initial concentration of Pb(II), $mg \cdot dm^{-3}$	Pair	t-coefficient calculated, $t_{crit for n = 5} = 2.776$	Statement
20.7	t_{1-2}	0.5535	Consistency
	t_{1-3}	4.4096	Inconsistency
	t_{2-3}	3.1978	Inconsistency
41.4	t_{1-2}	1.1003	Consistency
	t_{1-3}	0.3384	Consistency
	t_{2-3}	2.1792	Consistency
62.1	t_{1-2}	0.2266	Consistency
	t_{1-3}	1.9480	Consistency
	t_{2-3}	1.7479	Consistency
82.8	t_{1-2}	0.7347	Consistency
	t_{1-3}	2.7807	Inconsistency
	t_{2-3}	2.3712	Consistency
103.5	t_{1-2}	2.5341	Consistency
	t_{1-3}	12.4069	Inconsistency
	t_{2-3}	14.7265	Inconsistency

presented in **Table 8**, while **Table 9** contains results of the consistency testing.

As seen from **Table 9**, consistency of Pb removal is observed at each Pb-concentration for both the ultrasonic water bath and ultrasonic water bath containing Perwoll (t_{1-2}). A comparison of the ultrasonic water bath with dichloromethane extraction (t_{1-3}) gives consistency for only 2 concentrations, while inconsistency is estimated in 3 cases. On the contrary, the ultrasonic water bath containing Perwoll and dichloromethane extraction (t_{2-3}) shows 3 cases of consistency and 2 cases of inconsistency. It is obvious that under testing, the effect of the scouring procedures on sorption for each of the cations applied provides different results. However, in general, the results indicate that any of the scouring procedures tested based on the loss of mass cannot be rejected due to inconsistency (**Table 3**). The scatter of results observed under the scouring conditions is not abnormal as it is incidental to fibre thickness. It is well known that a sheep does not have the same wool thickness on the whole body, and it may typically vary from 18 to 40 μm. In the case of the same mass of wool being used in the washing experiment, a thinner fibre means a greater

surface, as well as the total amount of potential adhered contaminant (including lanoline) being used as a criterion. Our samples were taken from the raw sheep fleece randomly to imitate the situation in practice. Thus we have to admit considerable variations in the mass loss during the scouring as well as in the sorption efficiency examined in the experiments. A considerable scatter of results for tensile properties due to various fibre thicknesses was observed also for sheep wool irradiated by an electron beam [22], and such a result scatter can be perceived as a general feature.

Concerning the sorption experiments, except for the concentrations applied, the conditions for Cu(II), Zn(II) and Pb(II) sorption were the same for individual metal cations. Thus only small differences could be expected in the sorption percentage for the comparable experiments, if any. However, this is not so. Results of the cation sorption provide random data with no regularity (**Tables 4-9**). Considering the number of inconsistent cases from all possible cases (19 in total) following the t_{1-2}, consistency, 37% of them should be rejected. This portion involves only Cu(II) and Zn(II) almost equally, while Pb(II) shows all results are consist-

ent. Criterion t_{1-3} involves most of the inconsistences, giving about 74%, with the Cu(II) contribution being the largest. In contrast, criterion t_{2-3} indicates the least inconsistences for Cu(II) and Pb(II) and the most for Zn(II).

We consider three possible reasons for the variability. The first is the inhomogeneity in the fibre thickness, since it defines the surface area available for mutual contact. The second reason can be the slightly different surface polarity due to the dissimilar removal of lipids as indicated by the mass lost under scouring. The different sorption properties of the metal cations applied should be considered as the third source of variability. According to several authors [23], an increase in the atomic size means a greater sorption, because a metal with a large ionic radius is readily available for interaction with the sorbent. Some papers [24] confirm such an opinion as well. As Pb has the largest ionic radius among the cations tested, it provides more consistent data than Cu and Zn, which are of comparable ionic radii. Moreover the cations tested have a different tendency to create complex types with keratin. On the other hand, optimal sorption of metal cation is related to the pH value of the batch and surface charge on sorbent [25, 26]. Regarding the targeted use of wool as a sorbent potentially subsequently modified by certain procedures, this aspect was not taken into consideration in this study since the factors mentioned can vary sporadically. That is why any adjusting of the pH value in the batch was not carried out in the testing, and only the relative sorption efficiency eliminating other effects was considered. The variability in data consistency observed is most probably a combination of the factors mentioned that may not be comprehensive.

Although the differences in scouring (and partial removal) efficiency are not significant, the sorption efficiency of wool cleaned by dichloromethane seems to be the highest. However, the slightly lower efficiency of the water ultrasonic bath regarding sorption is highly compensated by the omission of the carcinogenic organic solvent under lanoline extraction or of off-loading waste water by detergents. In addition, the waste water without any chemicals can be used for lanoline salting or supplementary fertilising of agricultural land without off-loading it to a sewage treatment plant. It is assumed that relevant life cycle assessment and

economic cost analysis will prove that scouring without chemicals is environmentally friendlier and even cheaper.

■ Conclusions

The examination of sheep wool scouring for sorption purposes involved three procedures. The results of scouring using an ultrasonic water bath heated to 40 °C, the same bath with the addition of a commercial detergent, and Soxhlet extraction with dichloromethane were tested for consistency based on mass loss under the scouring. Using the Student's *t*-criterion, the scouring effects were considered as mutually consistent. The following targeted testing of Cu(II), Zn(II) and Pb(II) sorption from aqueous solutions by the scoured wool samples provided variable results indicating acceptable consistency of cations removal efficiency for scouring in a heated ultrasonic water bath with or without detergent. Sorption consistency was obtained in less than 50% of cases in dichloromethane extraction and both ultrasonic baths. Any clear coherence between the scouring used and sorption of the cations mentioned by the scoured wool could not be concluded. In summary, the using an ultrasonic water bath heated to 40 °C with no chemical additive provides the most suitable scouring procedure since the slightly lower sorption is compensated by other benefits. The omission of a detergent or carcinogenic solvent makes the ultrasonic process cleaner.

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47th **Textile Research Symposium 2019**
June 17-19, 2019, Liberec, Czech Republic