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The Study of Control Parameters for Some Divalent Metal Cations Sorption by Recycled Wool-based Nonwoven Material

[1]Maja Radetić, [1]Darinka Radojević, [1]Vesna Ilić, [1]Dragan Jocić, [2]Dragan Povrenović,
[3]Nevena Puač, [3]Zoran Lj. Petrović and [1]Petar Jovančić

[1]Textile Engineering Department, Faculty of Technology and Metallurgy,
Karnegijeva 4, 11000 Belgrade, Serbia
[2]Department of Environmental Engineering, Faculty of Technology and Metallurgy,
Karnegijeva 4,11000 Belgrade, Serbia
[3]Institute of Physics, Pregrevica 118, 11080 Zemun, Serbia

Abstract: The objective of this study was to highlight the influence of control parameters (initial concentration, temperature and pH) on lead, copper, zinc and cobalt cations uptake by recycled wool-based nonwoven material. The temperature and pH increase positively affected the sorption of metal cations. An increase in initial concentration of metal cations in the solution brought about increase in metal cation uptake, but the percentage of adsorbed metal cations decreased. Either as an untreated or modified (low-temperature air plasma, biopolymer chitosan and hydrogen peroxide treated) material, investigated sorbent showed good ability to bind all studied metal cations in the following order: $Pb^{2+}>Cu^{2+}>Zn^{2+}>Co^{2+}$.

Key words: Adsorption, lead, copper, zinc, cobalt

Introduction

In addition to many harmful and toxic substances present in industrial effluents, high concentration of metal cations is of great concern from environmental point of view. A wide range of industries (mining, metal processing, electroplating, electronics, chemical processing, textile, etc.) release into the environment various metals in amounts, which can pose a risk to human health. Therefore, potentially toxic and sometimes carcinogenic heavy metal ions must be removed from effluent before their release. The composition of effluents dictates the selection of wastewater treatment. In spite of many disadvantages, adsorption is still commonly applied for the purification of effluents and many different sorbents are currently available (Laing, 1991, Masri *et al.*, 1974). Keeping in mind wool ability to bind metal cations and its biodegradability, we have developed recycled wool-based nonwoven material with multifunctional sorption properties, which can be used for the removal of heavy metal cations and dyes from water medium as well as for oil spill cleanup (Radetić *et al.*, 2003a,b, 2005).

Our previous study indicated that recycled wool-based nonwoven material exhibited great ability to bind lead cations (Radetić *et al.*, 2003a). In order to improve its sorption properties, material was modified with low-temperature plasma and/or biopolymer chitosan, which is established as very efficient, multifunctional and relatively cheap biosorbent (Kulak *et al.*, 1999; Rhee *et al.*, 1998; Eiden *et al.*, 1980). Intrigued with results obtained, we decided to explore the possibility of application of hydrogen peroxide treatment to facilitate the binding of chitosan to wool and hence, improve the sorption capacity. In order to elucidate the further potentials of the material our research was extended to copper, zinc and cobalt cations. The study on sorption kinetics revealed that metal cation uptake

Corresponding Author: Maja Radetić, Textile Engineering Department, Faculty of Technology and Metallurgy,
Karnegijeva 4, 11000 Belgrade, Serbia

is strongly affected by sorption time (Djordjević *et al.*, 2005a) as well as that material possesses excellent selectivity due to different sorption rate of metal cations studied (Radetić *et al.*, 2003c). The aim of this study was to consider the influence of some control parameters (initial concentration, temperature and pH) on lead, copper, zinc and cobalt cations uptake by recycled-wool based nonwoven material.

Materials and Methods

Materials

The recycled wool-based nonwoven material (78% wool/22% polyester) was produced from secondhand military knitted pullovers of the constant quality and characteristics. A procedure for the production of recycled wool-based nonwoven material is described in literature in detail (Radetić *et al.*, 2003a).

Chitosan (CHT) (Vanson, Redmond - WA, USA), with a viscosity of 16 mPa·s and deacetylation degree of 88.6% was used without further purification for nonwoven material treatment for investigation on sorption of lead, cooper and zinc ions. Chitosan ChitoClear® (Primex, USA) with a viscosity of 102 mPa·s and deacetylation degree of 96% was applied for the study on cobalt ions. Acetic acid (96%, Lach-Ner, Czech Republic) was used for the preparation of chitosan solution. $Pb(NO_3)_2$ (MP Hemija, Serbia), $Cu(NO_3)_2 \cdot 3H_2O$ (Centrohem, Serbia, $Zn(NO_3)_2 \cdot 6H_2O$ (Centrohem, Serbia) and $Co(NO_3)_2 \cdot 6H_2O$ (Centrohem, Serbia) a were used for the investigation on cations sorption. 0.0100 M KNO_3 (Centrohem, Serbia) was applied as a supporting electrolyte. For hydrogen peroxide treatment, H_2O_2 (30 vol%, Centrohem, Serbia), $Na_4P_2O_7 \cdot 10H_2O$ (Kemika, Croatia) and NH_3 (aq) (Zorka, Serbia) were used.

Treatments

Low-Temperature Plasma (LTP) treatment was carried out in capacitively-coupled, radio-frequency (13.56 MHZ) air induced plasma (Radetić, 1998). Treatment time was 50 s, pressure of 0.27 mbar with the power supply maintained at a constant level of 100 W. LTP treatment of material was conducted in the Gaseous Electronics Laboratory of the Institute of Physics in Zemun (Serbia).

Two different procedures for the treatment with biopolymer chitosan were applied. The procedure A (CHT A) was based on immersion of samples in 0.1% CHT solution (liquor ratio 30:1) which were then shaken for 6 h, squeezed out through laboratory squeeze rolls and dried at room temperature (Radetić, 2003). Subsequently, the samples were treated with 5% solution of NH_3 (liquor ratio 30:1) for 10 minutes at room temperature in order to enhance the binding of chitosan to wool. Squeezed samples were dried at room temperature, washed with tap water and dried again at room temperature. 0.1% solution of chitosan was prepared according to the following procedure: 1.00 g of chitosan was thoroughly stirred with 1 mL of acetic acid and distilled water which was added six times in portions of 2 mL. After homogenisation, the formed gel stayed overnight. Afterwards, volumetric flask of 1 L was filled up with distilled water and solution was applied to samples.

The procedure B (CHT B) was based on immersion of samples in 0.3% CHT (liquor ratio 30:1) which were shaken for 20 min, squeezed out through laboratory squeeze rolls and dried at room temperature. Afterwards, they were washed with tap water and dried at room temperature. 0.3% solution of chitosan was prepared according to the following procedure: 3.00 g of chitosan was stirred in 0.4% acetic acid. Volumetric flask of 1 L was filled up with 0.4% acetic acid and solution stayed overnight ready for the application to material.

Hydrogen peroxide treatment (H_2O_2, 20 mL L^{-1}; $Na_4P_2O_7$, 1.5 g L^{-1} and NH_3 (aq) 2.5 mL L^{-1}) was done in static conditions (without shaking). Samples were treated in the solution for 1 h (liquor ratio 30:1) at 70°C and pH 9.40, washed with tap water and dried at room temperature.

Methods

The metal cation uptake (q, mg g^{-1}) was determined as a difference between the initial concentration of metal cations in the solution (C$_0$, mg L^{-1}) and the final concentration of metal cations in the solution (C$_f$, mg L^{-1}) (Eq. 1). Atomic Absorption Spectrometer (AAS 403, Perkin Elmer, USA) was used for determination of the concentration of metal cations in the solution.

$$q = \frac{(C_0 - C_f) \cdot V}{m} \tag{1}$$

The symbol V presents the solution volume (L) and m is the mass of sorbent material (g).
The percentage of metal cations adsorbed (MCA,%) was determined as (Eq. 2):

$$MCA = \frac{q}{q_{max}} \cdot 100 \quad [\%] \tag{2}$$

where:
q$_{max}$ maximum possible metal cation uptake (mg g^{-1}), which is calculated as (Eq. 3):

$$q_{max} = \frac{C_0 \cdot V}{m} \tag{3}$$

The influence of following parameters on sorption has been studied:

- Initial metal cation concentration-1.00 g of material was shaken in 50 mL of metal salt solution of different concentrations (C$_0$ = 50, 100, 250 and 500 mg L^{-1}) at pH 4.50 for 3 h;
- Temperature-1.00 g of material was shaken in 50 mL of metal salt solution (C$_0$ = 100 mg L^{-1}) at pH 4.50 for 1 h in water bath WB14 (Memmert, Germany) supplied with shaking device. Temperature of the solutions was maintained at 20, 40 and 60°C;
- pH-1.00 g of material was shaken in 50 mL of metal salt solution (C$_0$ = 100 mg L^{-1}) for 1 h. Appropriate initial pH values of the solutions (pH$_0$) were adjusted with 1.00 M HNO$_3$, 0.100 M HNO$_3$ and 0.100 M KOH. After the sorption, final pH values of the solutions (pH$_f$) were measured using an Inolab 730 (WTW, Germany) pH-meter. Initial pH values were adjusted to 2.50, 3.50 and 4.50.

Results and Discussion

The Influence of Initial Concentration

The results of sorption kinetics (Djordjević *et al.*, 2005a,b) demonstrated that approximately 87-99% Pb^{2+} ions, 52-97% Cu $^{2+}$ions, 33-100% Zn $^{2+}$ions and 86-100% Co $^{2+}$ions were already removed within 3 h, depending on the sorbent treatment. This was the reason to select the sorption time of 3 h for a study on influence of initial concentration on metal cation uptake. The influence of initial concentration on Pb^{2+}, Cu^{2+}, Zn^{2+} and Co^{2+} ion uptake for differently treated samples is demonstrated in Fig. 1.

At low initial Pb^{2+} ion concentrations (<100 mg L^{-1}), there was a negligible difference between differently treated samples and uptake from the solution of initial concentration of 50 mg L^{-1} was almost complete. However, at higher initial concentrations treated samples started to demonstrate their
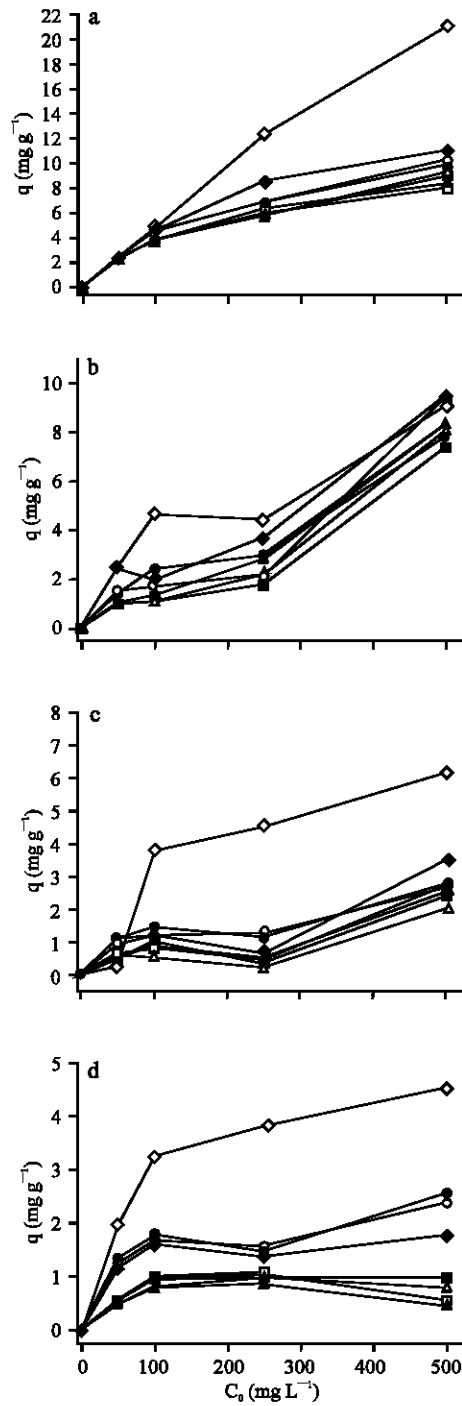
Fig. 1: Influence of initial concentration on uptake of: (a) Pb^{2+} ions, (b) Cu^{2+} ions, (c) Zn^{2+} ions, (d) Co^{2+} ions (T = 20°C, pH_0 = 4.50, t = 3 h). □ untreated; ■ LTP; ○ CHT A; ● LTP+CHT A; △ CHT B; ▲ LTP+CHT B; ◇ H_2O_2; ◆ H_2O_2+CHT B

advantages. Apparently, all treated samples adsorbed higher amounts of Pb^{2+} ions than untreated sample at initial concentration of 500 mg L^{-1}. This increase was considerably more pronounced in case of CHT A, LTP+CHT A, H_2O_2+CHT B and particularly H_2O_2 treated samples. Curves related to LTP, CHT B and LTP+CHT B treated samples almost overlapped at initial concentrations between 50-250 mg L^{-1}, but slight difference in Pb^{2+} ion uptake occurred at initial concentration of 500 mg L^{-1}. Achieved results were in good correlation with results corresponding to sorption kinetics (Djordjević *et al.*, 2005a,b).

Figure 1b revealed the same shape of all curves for the sorption of Cu^{2+} ions indicating the same function of Cu^{2+} ion uptake on initial concentration independently on sample studied. Untreated and LTP treated samples behaved identically in the whole range of investigated initial concentrations of Cu^{2+} ions. Differences in Cu^{2+} ion uptakes between tested samples can be distinguished already at initial concentration of 50 mg L^{-1}. It is obvious that sorption was not significantly affected by initial Cu^{2+} ion concentrations in the interval ranging from 50-250 mg L^{-1}. The rise of Cu^{2+} ion uptake was observed at initial concentration of 500 mg L^{-1}. H_2O_2 treated sample exhibited better sorption properties compared to untreated sample, but the contribution of H_2O_2 treatment was far away from the results obtained for Pb^{2+} ions.

Table 1: The percentage of metal cations adsorbed (T = 20 °C, pH_0 = 4.50, t = 3 h)

	MCA, (%)			
	Pb^{2+}	Cu^{2+}	Zn^{2+}	Co^{2+}
C_0 = 50 mg L^{-1}				
Untreated	92.2	40.0	25.5	22.3
LTP	90.6	60.4	19.8	22.0
CHT A	97.8	60.4	38.0	50.3
LTP+CHT A	99.8	56.0	23.7	52.9
CHT B	92.8	42.6	42.8	20.4
LTP+CHT B	90.6	39.6	21.4	20.3
H_2O_2	100	99.4	9.88	78.3
H_2O_2+CHT B	99.0	99.2	44.3	46.3
C_0 = 100 (mg L^{-1})				
Untreated	77.8	22.8	16.6	19.7
LTP	76.0	22.4	20.6	19.2
CHT A	91.6	34.6	23.8	33.5
LTP+CHT A	92.4	48.6	29.4	35.4
CHT B	76.4	31.1	10.6	16.1
LTP+CHT B	78.6	27.8	17.6	15.7
H_2O_2	99.4	93.2	75.4	64.6
H_2O_2+CHT B	94.6	40.4	24.6	32.5
C_0 = 250 (mg L^{-1})				
Untreated	51.2	14.2	3.54	8.43
LTP	47.5	14.2	3.06	8.14
CHT A	54.8	17.4	10.2	12.4
LTP+CHT A	54.6	23.8	9.20	11.9
CHT B	47.1	18.1	2.04	7.96
LTP+CHT B	48.2	22.6	4.08	6.97
H_2O_2	99.7	35.5	36.0	30.5
H_2O_2+CHT B	68.8	29.7	5.10	11.0
C_0 = 500 (mg L^{-1})				
Untreated	32.2	29.7	11.0	2.23
LTP	35.8	29.7	9.68	3.92
CHT A	40.9	37.7	10.7	9.56
LTP+CHT A	39.5	31.3	11.2	10.2
CHT B	37.1	32.2	8.16	3.20
LTP+CHT B	33.6	33.6	10.2	1.92
H_2O_2	83.9	36.4	24.1	18.1
H_2O_2+CHT B	44.2	38.1	14.1	7.11

As it appeared in study on sorption kinetics (Djordjević *et al.*, 2005a,b), sorption behaviour of Zn^{2+} ions differed from that noticed in case of Pb^{2+} and Cu^{2+} ions. Only H_2O_2 and H_2O_2+CHT B treatments led to an increase in Zn^{2+} ion uptake. H_2O_2 treated sample showed completely different trend of sorption than other samples. Zn^{2+} ion uptake for CHT A and LTP+CHT A treated samples were higher in comparison with untreated sample at initial concentrations between 50-250 mg L^{-1}, but at 500 mg L^{-1} there was almost no difference between these samples and corresponding points overlapped. LTP, CHT B and LTP+CHT B treated samples seemed to bind less Zn^{2+} ions than untreated sample at initial concentrations between 250-500 mg L^{-1}.

The rise of initial concentration brought about increase in Co^{2+} ion uptake in case of CHT A, LTP+CHT A, H_2O_2 and H_2O_2+CHT B treated samples. This effect was more evident at higher concentrations (500 mg L^{-1}). On the contrary, sorption on other samples was insignificantly affected by initial concentration. Unexpectedly, at concentration of 500 mg L^{-1} slight decrease in Co^{2+} ions uptake was observed.

Obviously, the increase in initial concentration brought about rise of metal cation uptake. Sheffield and Doyle found that the Cu^{2+} uptake increased as the applied amount of Cu^{2+} ions in solution increased (Sheffield and Doyle, 2005). Similar trend was established in the study on sorption of copper by mohair (Guthrie and Laurie, 1968). The percentage of metal cation adsorbed as a function of initial concentration of the metal cations in the solution is shown in Table 1. Although uptake of metal cations increased, the percentage of metal cations adsorbed by untreated and differently treated materials decreased. Some deviations occurred in case of Cu^{2+} and Zn^{2+} ions for the concentrations of 250 and 500 mg L^{-1}. Reduction of the fraction of metal cations adsorbed indicates that higher amounts of metal cations are left in the solution (Sheffield and Doyle, 2005).

The Influence of Temperature

The influence of temperature on Pb^{2+}, Cu^{2+}, Zn^{2+} and Co^{2+} ion uptake for differently treated samples is presented in Fig. 2. Sorption time was chosen to be 1 h as it is indicated that longer heating of the solution leads to an increase in copper uptake, but this is mainly attributed to the wool hydrolysis (Sheffield and Doyle, 2005). Temperature showed a strong influence on the sorption of all investigated metal cations. The higher the temperature, the higher was the metal cation uptake. However, the exceptions occurred in case of sorption of Co^{2+} ions on untreated, LTP and CHT B treated samples where the rise of temperature caused a slight decrease in metal cation uptake.

The Influence of pH

The influence of final pH value (after 1 h of sorption) on Pb^{2+}, Cu^{2+}, Zn^{2+} and Co^{2+} ion uptake for differently treated samples is presented in Fig. 3. As expected, the higher the pH, the higher was the metal cation uptake. The solubility products of $Pb(OH)_2$, $Cu(OH)_2$, $Zn(OH)_2$ and $Co(OH)_2$ at 25°C are 2.8×10^{-16}, 1.6×10^{-19}, 4.5×10^{-17} and 2.5×10^{-16}, respectively. According to solubility calculation, precipitation of hydroxides for concentrations of 100 mg L^{-1} should appear above pH 7.88, 6.00, 7.23 and 7.58, respectively. The results from Fig. 3 pointed out that final pH values were below the calculated values suggesting that no precipitation could be anticipated. There was also no visible precipitate in the solution.

At lower pH material is positively charged i.e., end and side amino groups of the wool protein chains are protonated. At higher pH sorption was enhanced as the wool became negatively charged and no repulsion of metal cations like at acidic conditions occurred. Thus, at higher pH carboxylate groups that are the main sites for binding of metal cations (Hemrajani and Nawani, 1967; Kokot and Feughelman, 1972) existed as anions.
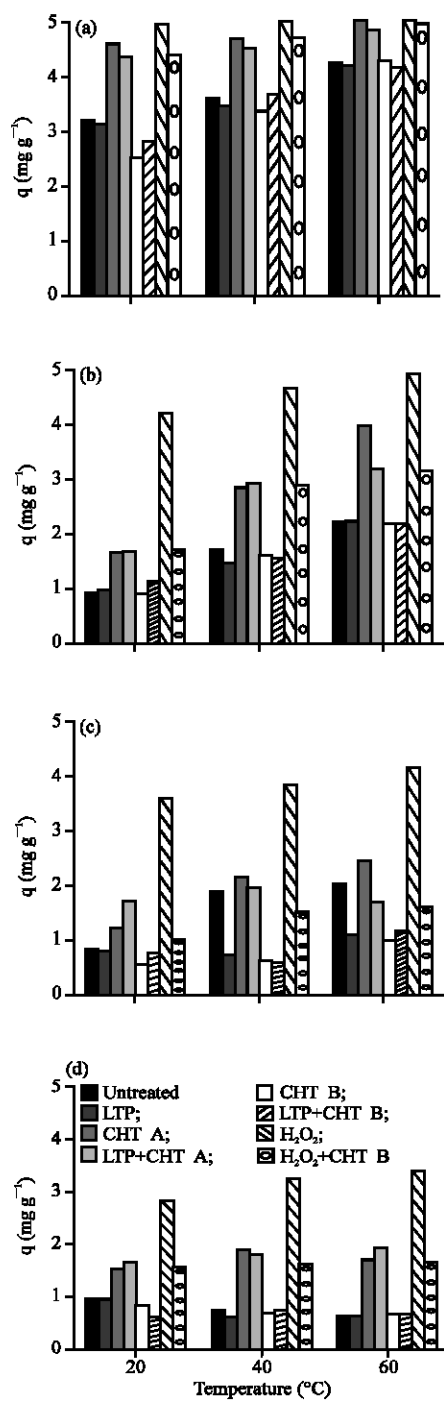
Fig. 2: Influence of temperature on uptake of: (a) Pb^{2+} ions, (b) Cu^{2+} ions, (c) Zn^{2+} ions, (d) Co^{2+} ions ($C_0 = 100$ mg L^{-1}, $pH_0 = 4.50$, t = 1 h)
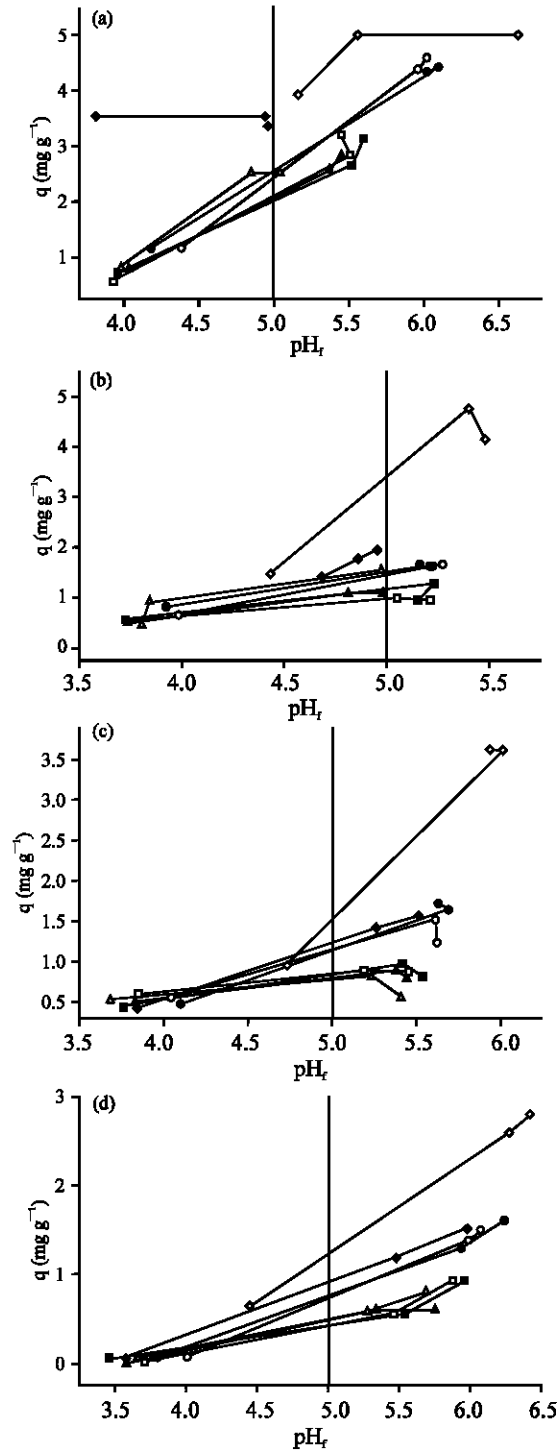
Fig. 3: Final pH vs. uptake of: (a) Pb^{2+} ions, (b) Cu^{2+} ions, (c) Zn^{2+} ions, (d) Co^{2+} ions (T = 20 °C, C_0 = 100 mg L^{-1}, t = 1 h) □ Untreated; ■ LTP; ○ CHT A; ● LTP+CHT A; Δ CHT B; ▲ LTP+CHT B; ◇ H_2O_2; ◆ H_2O_2+CHT B

Table 2: The increase in pH of the solution after the sorption of heavy metal cations (T = 20°C, C_0 = 100 mg L^{-1}, t = 1 h)

	ΔpH^*			
	Pb^{2+}	Cu^{2+}	Zn^{2+}	Co^{2+}
pH_0 = 4.50				
Untreated	0.95	0.71	0.95	1.38
LTP	1.10	0.65	1.04	1.46
CHT A	1.52	0.71	1.12	1.58
LTP+CHT A	1.52	0.66	1.13	1.74
CHT B	0.54	0.47	0.91	1.19
LTP+CHT B	0.95	0.48	0.94	1.26
H_2O_2	2.13	0.98	1.43	1.92
H_2O_2+CHT B	0.46	0.45	1.01	1.48
pH_0 = 3.50				
Untreated	2.01	1.55	1.69	1.97
LTP	2.02	1.73	1.92	2.04
CHT A	2.46	1.77	2.11	2.49
LTP+CHT A	2.60	1.72	2.19	2.44
CHT B	1.35	0.34	1.73	1.78
LTP+CHT B	1.87	1.31	1.88	1.84
H_2O_2	2.06	1.90	2.51	2.78
H_2O_2+CHT B	1.44	1.36	1.76	1.98
pH_0 = 2.50				
Untreated	1.43	1.23	1.35	1.22
LTP	1.46	1.22	1.26	0.96
CHT A	1.88	1.48	1.54	1.51
LTP+CHT A	1.68	1.42	1.60	1.30
CHT B	1.48	1.30	1.18	1.16
LTP+CHT B	1.53	1.23	1.33	1.08
H_2O_2	2.66	1.93	2.23	1.95
H_2O_2+CHT B	1.31	2.18	1.34	1.08

$^*\Delta pH = pH_f\text{-}pH_o$

The pH value of 5.00 was chosen for the comparison of our results with literature data. It has been established that metal cation uptake by raw wool increases in the following order: $Pb^{2+}>Cu^{2+}>Zn^{2+}>Co^{2+}$ (Masri *et al.*, 1974; Weltrowski *et al.*, 1995). Present study confirmed the same order in case of recycled wool as well.

It is well known that investigated cations in water can undergo hydrolysis which basically depends on the concentration of metal ions in solution, the pH and the type of anions present in solutions (Kratochvil *et al.*, 1998). The hydrolysis of Me^{2+} ion (Me^{2+} = Pb^{2+}, Cu^{2+}, Zn^{2+}, Co^{2+}) can be written in simplified form as:

$$Me^{2+} + H_2O \Leftrightarrow MeOH^+ + H^+ \tag{4}$$

Obviously, monovalent cations $Me(OH)^+$ and protons are generated causing the increase in acidity of the solution. If Me^{2+} is bound to the wool, reaction (Eq. 4) is shifted towards left and solution becomes more alkaline (Kratochvil *et al.*, 1998). However, it must be kept in mind that pH of a solution may be strongly influenced by release and/or uptake of protons.

The results in Table 2 demonstrated that pH values after 1 h of sorption (pH_f) increased considerably compared to initial pH_0 values of 2.50, 3.50 and 4.50. Thus, it is suggested that a rise of pH of the solutions is likely due to the sorption of metal cations in the form of Me^{2+}, but the parallel release and uptake of protons should not be neglected. In other words, two competitive processes govern pH changes: the sorption of metal cations and release and/or uptake of protons.

Conclusions

Recycled wool-based nonwoven material is an efficient sorbent for lead, copper, zinc and cobalt cations. The best adsorbed metal is lead, followed by copper, zinc and cobalt. Sorbent possesses satisfactory sorption capacity even as an untreated material. LTP treated samples exhibited similar sorption behaviour as untreated samples, whereas chitosan and particularly hydrogen peroxide treatments brought about significant increase in uptake of all investigated metal cations. The positive effect of different treatments became more obvious at higher initial concentrations of metal cations in the solution.

An increase in initial metal cation concentration caused the rise of metal cation uptake, but the percentage of adsorbed metal cations decreased. A rise of temperature and pH positively affected the sorption of metal cations. The results indicated that sorption of metal cations is strongly influenced by initial concentration, temperature and pH, suggesting that appropriate adjustment of solution parameters can provide optimum sorption, which can be utilized in real systems for the efficient removal of metal cations from effluents.

Acknowledgment

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