Journal of Environmental Science and Technology

ISSN 1994-7887

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OPEN ACCESS Journal of Environmental Science and Technology

ISSN 1994-7887
DOI: 10.3923/jest.2017.107.119

Research Article
Removal of Heavy Metals from Aqueous Solution by Adsorption using Livestock Biomass of Mongolia

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Abstract
Background and Objective: There is damaging pollution of heavy metals around the mining area in Mongolia. Therefore, it is a quite important mission to develop a removal and remediation methods of heavy metals. The objective of this study was the preparation of adsorption materials based on biomasses of livestock and their removal performance of heavy metals from aqueous solution. **Materials and Methods:** The livestock biomasses, which are sheep manure, wool and fat are used to adsorb Cu (II), Pb (II) and Cd (II). The sheep wools treated with the alkaline solutions were also used for the adsorption. The studies of materials morphology were made by SEM and EDAX analysis. The functional groups of the alkaline treated wool were determined by FTIR. The adsorption studies were made under various conditions, such as effects of pH, initial heavy metal concentration and NaOH treatment time. **Results:** In the morphology studies, a sheep wool cuticle was broken after treatment of NaOH and Na_2S. Fourier Transform Infrared spectroscopy data defined changes of treatment of wool by alkaline. The functional groups of alkaline treated wool were determined high intensity, comparing to the untreated wool. The adsorption amounts of heavy metals of Cu (II), Pb (II) and Cd (II) were increased after the alkaline treatment. Thus adsorption amount increased with increasing NaOH treatment time 1-20 h. **Conclusion:** It was concluded that the wool adsorbent treated by Na_2S and NaOH would use as an adsorbent for removal of heavy metals from contaminated rivers.

Key words: Adsorption, heavy metals, livestock biomass, sheep wool, adsorbent, alkaline treatment

Citation: Solongo Enkhzaya, Koichiro Shiomori and Bolormaa Oyuntsetseg, 2017. Removal of heavy metals from aqueous solution by adsorption using livestock biomass of Mongolia. J. Environ. Sci. Technol., 10: 107-119.

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Competing Interest: The authors have declared that no competing interest exists.

Data Availability: All relevant data are within the paper and its supporting information files.

INTRODUCTION

As increasing activity of the mining industry in Mongolia, it has been reported a shattering pollution of heavy metals around the mining area[1–4]. Therefore, it is a quite important mission to develop the removal and remediation methods of heavy metals with both high efficiency and low-cost. This study describes heavy metals adsorption performances of livestock biomasses, such as sheep manure, fat and wool and chemical treatment of sheep wool to increase adsorption performance of heavy metals from aqueous solution.

Mongolia has ample natural resources of coal, copper, gold and oil. Boroo gold mining area and Erdenet copper mining area are located in the Northern part of Ulaanbaatar, the capital city of Mongolia. Tamsag bulag petroleum industry is located in Dornod province about 856 km East part of Ulaanbaatar city. Oyu-Tolgoi gold-copper mining area and TavanTolgoi coal mining area are located in Umnugovi province about 235 km South part of Ulaanbaatar city, respectively. Small-scale gold mining area, there has called Ninja (people) are lives for extracting gold and sells to the speculators. They are using a very dangerous method such as mercury method to extract gold from the soil illegally[5]. Around mining area, river and land were polluted by toxic substances and heavy metals. Rehabilitation is necessary to the environment after mining operations[2]. Mongolian mining area is contaminated by high amount of heavy metals and following studies were done in Mongolia. Heavy metal situation in Mongolia:

- Boroo River water containing high amount of Mn, Fe, Co and As (217 ppb) and river sediment containing more higher amount of As (1845 ppm) are cause of sedimentation process. Gatsuurt River water containing high amount of heavy metals such as Cr, Fe, Co, Ni, Cu, Zn, As, Cd and Sb. Amount of As was 24.0-61.7 ppb in 2010, but after one year analysis amount of As decreased by a half, 16.2-26.4 ppb in river water samples cause of increasing amount of water[2]
- Surface soil, groundwater and hair samples were analyzed around Boroo gold mining area and these samples containing high amount of heavy metals. Cu (109 ppm), Zn (146 ppm) as (167 ppm) and Pb (86 ppm) determined in soil samples. Mn, Fe and Ni amount determined higher than WHO standard in ground water sample and amount of Ca, Ti as and Sr in hair samples were higher than elements of healthy people hair samples[3]
- Tree bark, lichens and street dust samples were collected around Erdenet copper mining area and Cu, Mn and Zn are highly contained in lichen and street dust samples. Fe, Zn, Mn and Cu were determined in bark samples and heavy metal contents increasing to decreasing distance to the copper mining area[1]

Therefore, the development of removal and remediation methods for heavy metals with high effectiveness and low cost for the mining area in Mongolia is a quite important mission.

Mongolian animal husbandry is one of the major parts of the agriculture sector and a number of Mongolian livestock reached 51.9 million in 2014[6]. Almost half of the total livestock is sheep and herders got many wastes such as sheep manure, sheep wool and sometimes sheep fats from the animal husbandry.

Sheep manure is consisting of mainly protein, cellulose, hemicellulose and lignin[7]. The solid fraction mainly contains phosphoric compounds while the liquid fraction contains nitrogen compounds[7]. The sheep manure is reported to be a good adsorbent for divalent metals[8-10].

Sheep wool is comprised of 49% C, 17% N, 21% O, 6% H, 5% S and trace elements (Al, Ba, Co, Cu, Fe, Mn, Mo, Sr, Ti, Zn). The sheep wool has three major classes of hair proteins such as low sulfur keratin (intermediate filament components of the hair cortex), high sulfur protein (cysteine-rich components; part of the interfilamentous matrix of the cortex) and high glycine/tyrosine protein (interfilamentous matrix)[11]. The sheep wool contains α-keratin and it formed like a rope strand. The α-keratin amino acids contain high amount of cysteine and low amount of hydroxyproline and proline[12]. Cystine is the most reactive amino acid residue in keratin. Keratin molecules in wool are fairly stable whereas cross linking bond with disulfide bonds[13]. The wool applies to adsorb heavy metals such as Cu, Cr, Cd, Co, Hg, Al, Ni, Zn, Au, Ag and Pb from waste waters[14–19]. Sometimes alkaline treatment of the wool consumes breakage of the disulfide bonds[20], which expected to increase the adsorption capacity of heavy metals[21–25].

This study will describes the removal of the heavy metals from aqueous solution by adsorption materials of the livestock biomasses and also their adsorption properties were studied.

MATERIALS AND METHODS

This study carried out from October, 2014-September, 2016 in University of Miyazaki, Japan.

Materials: The chemical substances of sodium hydroxide, hydrogen chloride solution and heavy metal standards used in this study were purchased from Wako Pure Chemical

Fig. 1: Image of sheep manure of Mongolia used in this study

Fig. 2: Image of sheep wool of Mongolia used in this study

Industries Ltd (Japan). Sheep manure shown in Fig. 1, wool shown in Fig. 2 and fats were collected from animal husbandry of Erdenet (Mongolia), the Northern part of Ulaanbaatar city.

Methods

Preparation of the sheep wool, manure and fat: Mongolian livestock biomasses, sheep wool and sheep manure was washed with distilled water and dried at room temperature. The sheep fat got from sheep tail, was used as adsorbents after cutting into small pieces. Alkaline dissolution mechanism of the sheep wool described as the amino acid chains, were settled basically on the cross-linkages of cystine[11].

Na_2S treatment: The treatment of Na_2S is used to separate the wool from the hide in the industry for a long time. Hydrogen bond of the wool is broken by the treatment of sodium sulfide. Furthermore, disulfide bonds break while dispersion by sodium sulfide into the wool[12]. About 12.5 g sheep wool (1.33 wt%) was put into 500 mL Na_2S (0.5 M) solution (pH = 10-13) for 6 h at 30°C[26]. The wool was washed with distilled water and dried at room temperature:

$$2R\text{-}X+Na_2S\leftrightarrow R_2S+2NaX$$

NaOH treatment: Alkali treatment made cleavages of disulfide bond in the wool. Random coil and α-helix would be lost their structures in the sheep wool proteins, furthermore, hydrogen bonds cleaved by the alkali treatment. The adsorption capacities increased by increasing the treatment time of sodium hydroxide and heavy metals adsorbed on adsorptive sites of the alkaline treated wool[27]. About 6.0 g wool (0.56 wt%) put into 500 mL NaOH (0.1 M) solution for 1, 10 and 20 h at 50°C. The wool was washed with distilled water and dried[20].

$$R\text{-}S\text{-}S\text{-}R+OH^-\rightarrow R\text{-}SOH+R\text{-}S^-$$

Scanning Electron Microscope (SEM) and Energy Dispersive X-ray Spectroscopy (EDAX) analysis: The morphologies of alkaline treated sheep wool samples were analyzed after Cu, Pb, Cd adsorption by the Energy Dispersive X-ray spectroscopy where the Scanning Electron Microscopes is in operation.

Fourier Transform Infrared spectroscopy (FTIR) measurements: The sheep wool and the alkaline treated sheep wool samples were prepared and interfold into pellets using KBr sheet by the manual hand press. The prepared sheep wool samples measured with JASCO 6600 Infrared Raman Spectrometer. The wave number was 400-4000 cm^{-1}, the scan number was 80 and resolution was 4 cm^{-1}.

Heavy metals adsorption analysis: Adsorption analysis of Cu, Pb and Cd was made by the heavy metal solution with the adsorbent and shaking. Their adsorption performances were measured with ICP-AES. The adsorption capacity[28] of the heavy metal ions were calculated by Eq. 1:

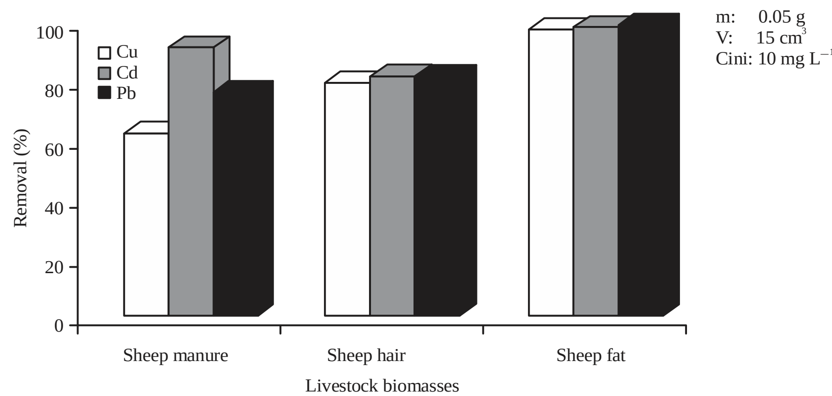


Fig. 3: Removal percentage of the heavy metals by livestock biomasses

$$q_e = \frac{C_0 - C_a}{m} \cdot V \quad (1)$$

q_e: Amount of heavy metal ions adsorbed at equilibriums per weight of adsorbent, (mg g^{-1})
C_0, C_a: Heavy metal ion concentration for initial and after adsorption, (mg L^{-1})
V: Volume of the solution, (L)
m: Amount of adsorbent, (g)

Langmuir adsorption isotherm: Langmuir isotherm models figure out the maximum adsorption capacity based on adsorption amount of adsorbent at isotherm condition. The Langmuir adsorption isotherm[29] is shown below:

$$q_e = \frac{Q_{max} K C_e}{1 + K C_e} \quad (2)$$

The linear form of the Langmuir equation[29] was given by Eq. 3:

$$\frac{1}{q_e} = \frac{1}{Q_{max}} + \frac{1}{K \cdot Q_{max}} \cdot \frac{1}{C_e} \quad (3)$$

Q_{max}: Maximum adsorption capacity (mg g^{-1})
C_e: Heavy metal concentration at equilibrium (mg L^{-1})
q_e: Equilibrium adsorption capacity (mg g^{-1})
K: Langmuir adsorption constant (L mg^{-1})

RESULTS

The livestock biomasses were determined as able to remove heavy metals from aqueous solution. Thus heavy metal adsorption amount increased after alkaline treatment of sheep wool.

Adsorption behavior of Mongolian biomass of livestock: Heavy metal removal percentage of the livestock biomasses represents that all the biomasses adsorbents are possible to adsorb heavy metals from aqueous solution as shown in Fig. 3. The removal efficiency of the heavy metals was determined as 62.28% and above in the sheep manure, 79.63% and above in the sheep wool, 97.68% and above in the sheep fat samples.

Effect of pH: The removal effects of the Cu, Pb, Cd by livestock biomasses at various initial pH values, 3, 5, 6 and 8 are shown in Fig. 4. The equilibrium pH was 5.87-7.20 after adsorption for the Cu solution and the removal efficiency of Cu was 60-88%. However, the lowest removal efficiency was observed for the sheep fat at an equilibrium pH at 3.5. Removal efficiency of Pb was 64.18% and above for Pb solutions except Pb removal by the sheep fat for pH_{eq} at 3.5. The high removal efficiency of Pb was obtained at pH_{eq} 5.9-7.55. Also, the removal efficiency of Cd defined as 81.36% and above, except for the Cd removal by the sheep fat.

Alkaline-treated sheep wool

Morphology analysis of alkaline treated sheep wool: The morphology change of sheep wool during the treatment was observed with SEM as shown in Fig. 5. The surface of wool fiber had cuticle cells as shown in Fig. 5a. The cuticle cells were broken by the NaOH treatment as shown in Fig. 5b. In Na_2S treatment, the wool surface was dissolved and changed to smooth as shown in Fig. 5c.

EDAX analysis: Heavy metals adsorbed on surface and cross section entirely in the alkaline-treated wool samples. Usually, heavy metals are adsorbed on protein rich keratin but the treatment of alkaline make high adsorption capacity. Wool cuticle cell was disrupted by alkaline treatment and cysteine would be reduced form cysteine. Also many different kinds of

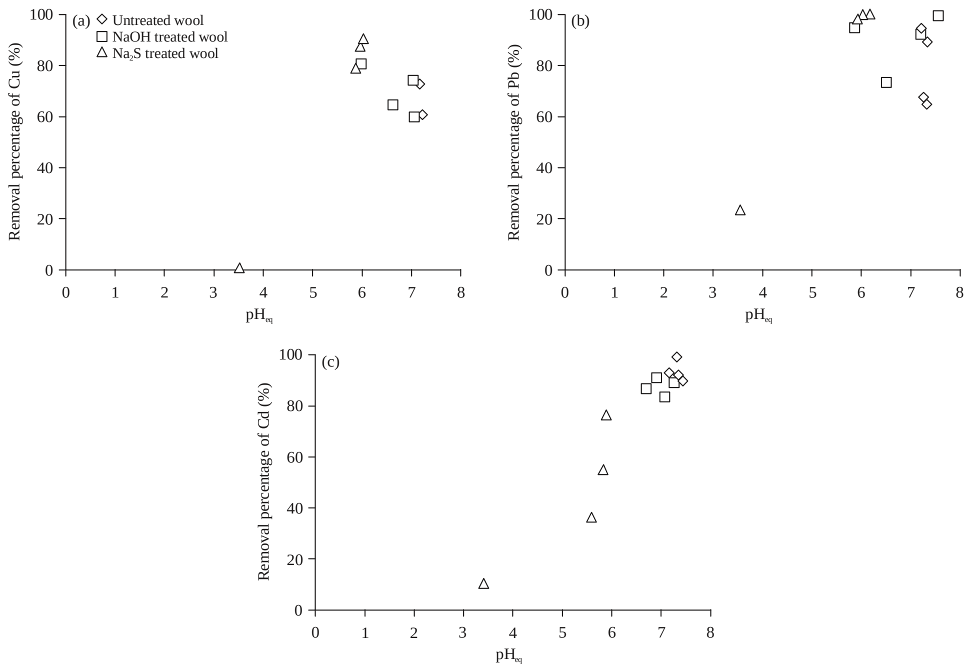


Fig. 4: Effect of pH on heavy metals removal by the sheep manure, wool and fat (a) Cu, (b) Pb and (c) Cd

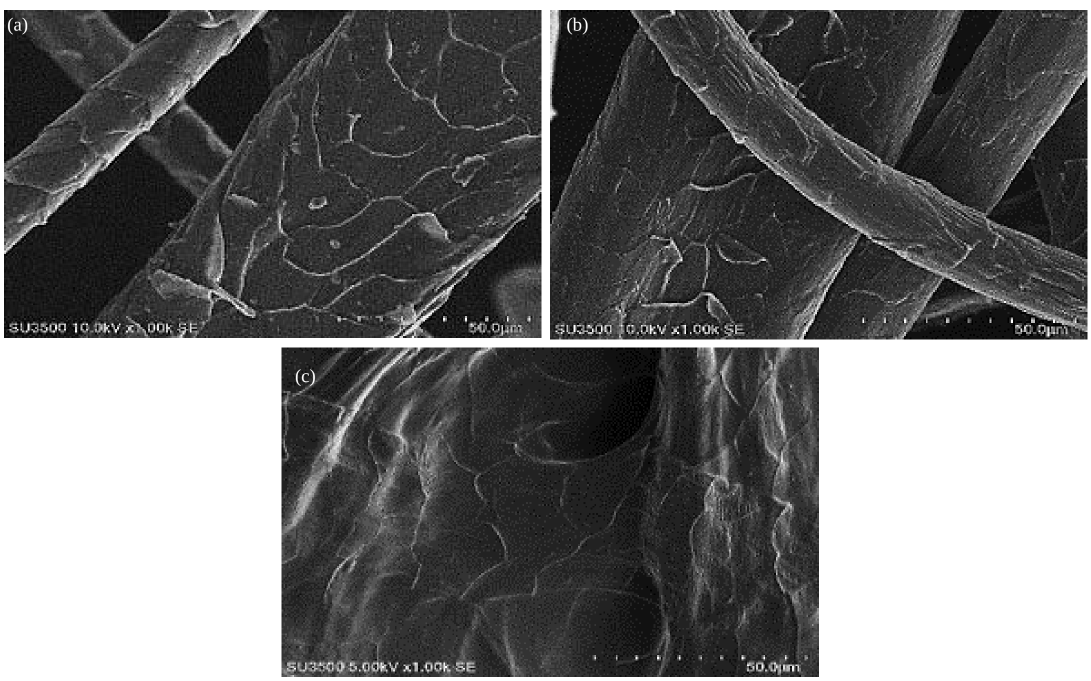


Fig. 5(a-c): Morphology of the wool surfaces (a) Untreated sheep wool, (b) NaOH treated sheep wool and (c) Na_2S treated sheep wool

active groups formed during the alkaline-treatment of the sheep wool. Heavy metal ions attracted to these active groups during the adsorption.

After adsorption of the heavy metals, all of the NaOH-treated wool samples mainly comprised of C, N, O, Na, Sr and S elements. Wool contained a quite little amount of Sr and also Sr amount observed by EDAX analysis[11]. The amount of heavy metals in the surface of the NaOH treated sheep wool samples were defined as 4.15% of Cu, 2.63% of Pb and 2.47% of Cd as shown in Fig. 6(a-f). After the adsorption of the heavy

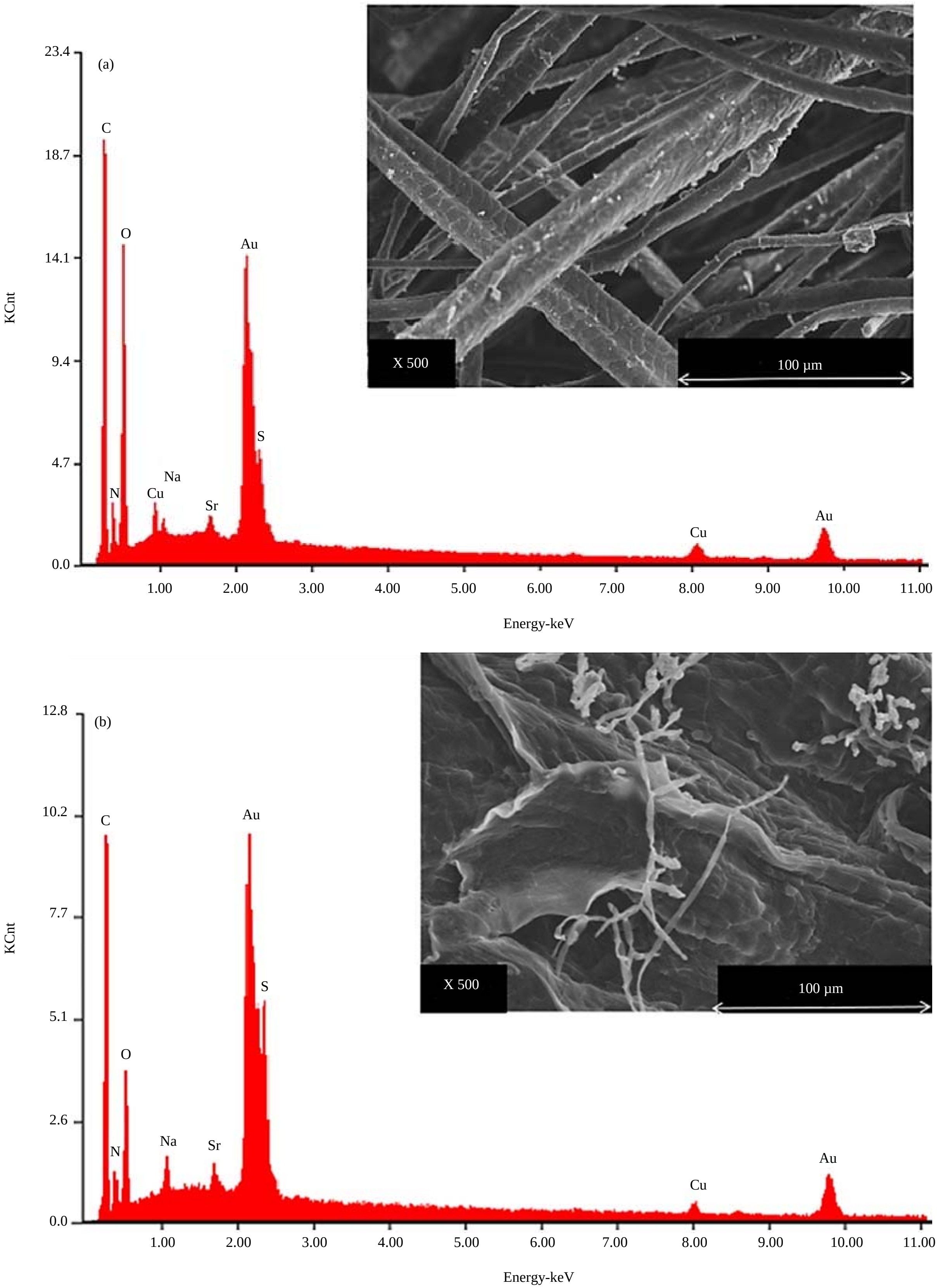
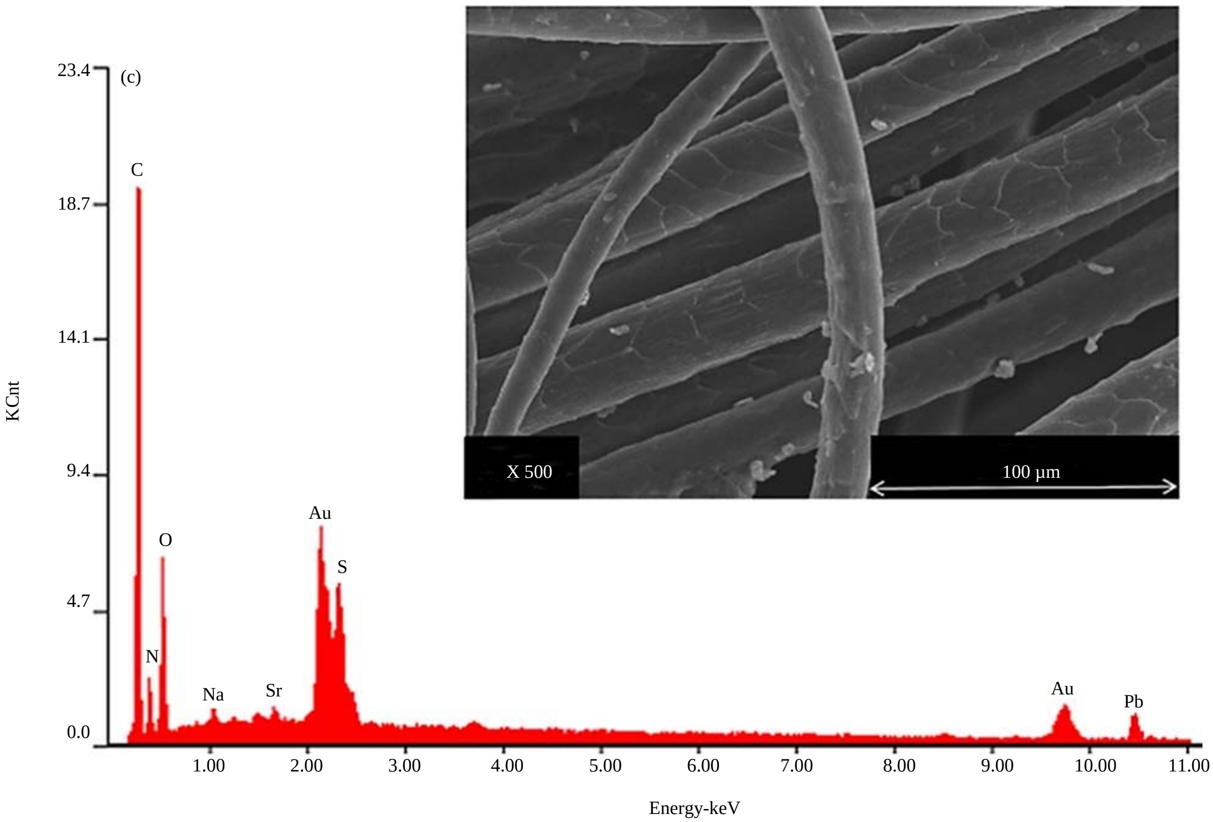
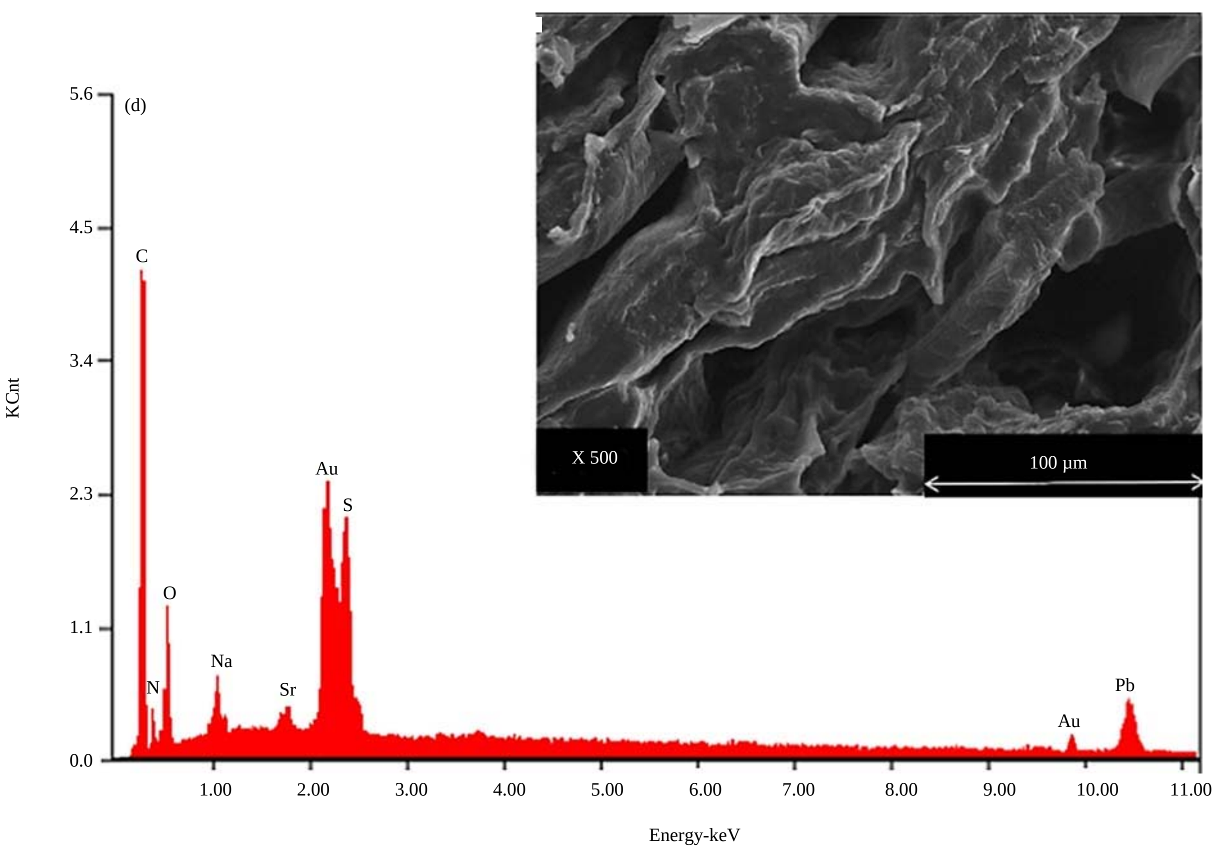


Fig. 6(a-f): Continue





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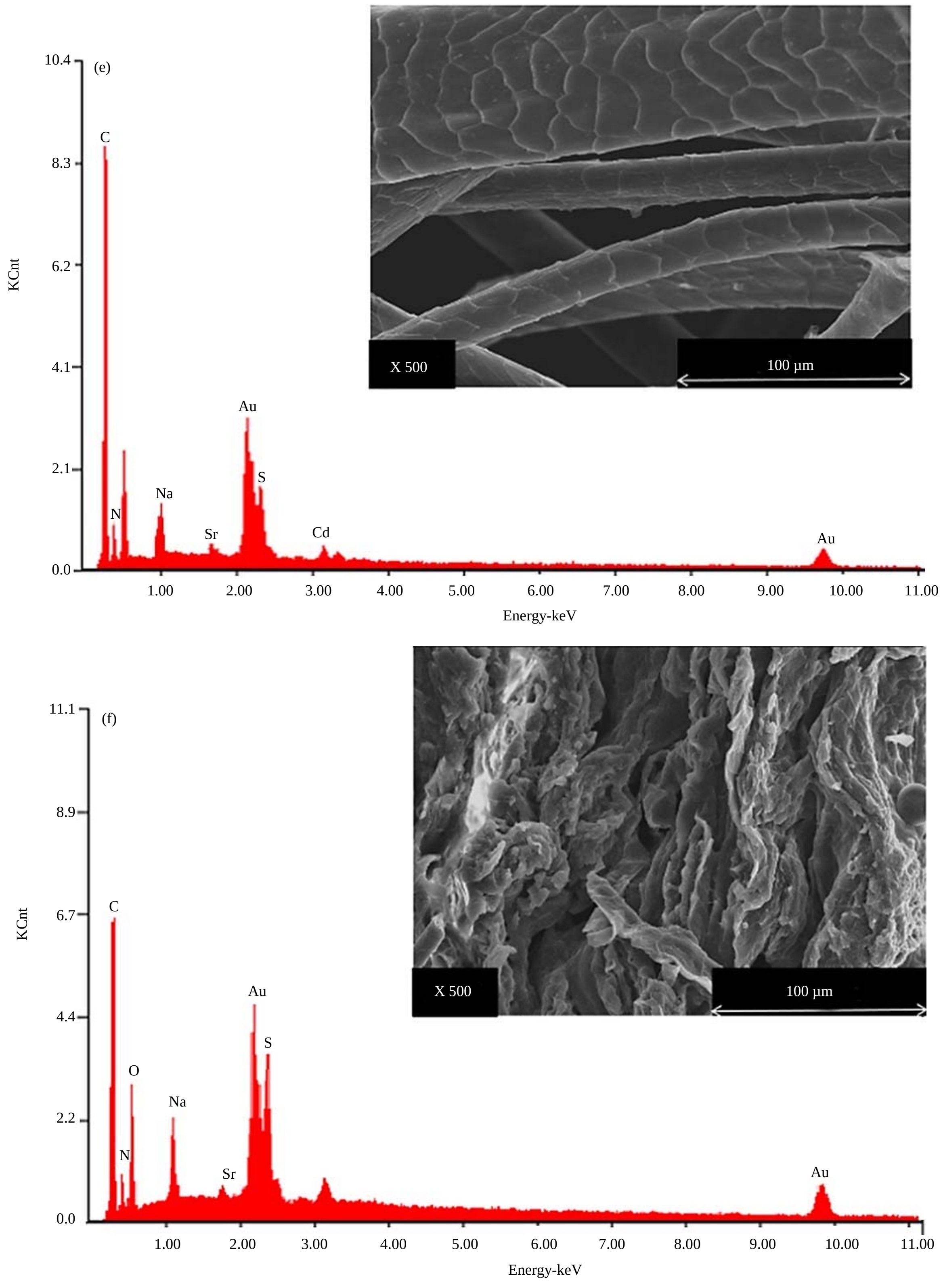

Fig. 6(a-f): EDAX analysis of the alkaline treated wools (a) NaOH treated wool after adsorption of Cu, (b) Na_2S treated wool after adsorption of Cu, (c) NaOH treated wool after adsorption of Pb, (d) Na_2S treated wool after adsorption of Pb, (e) NaOH treated wool after adsorption of Cd and (f) Na_2S treated wool after adsorption of Cd

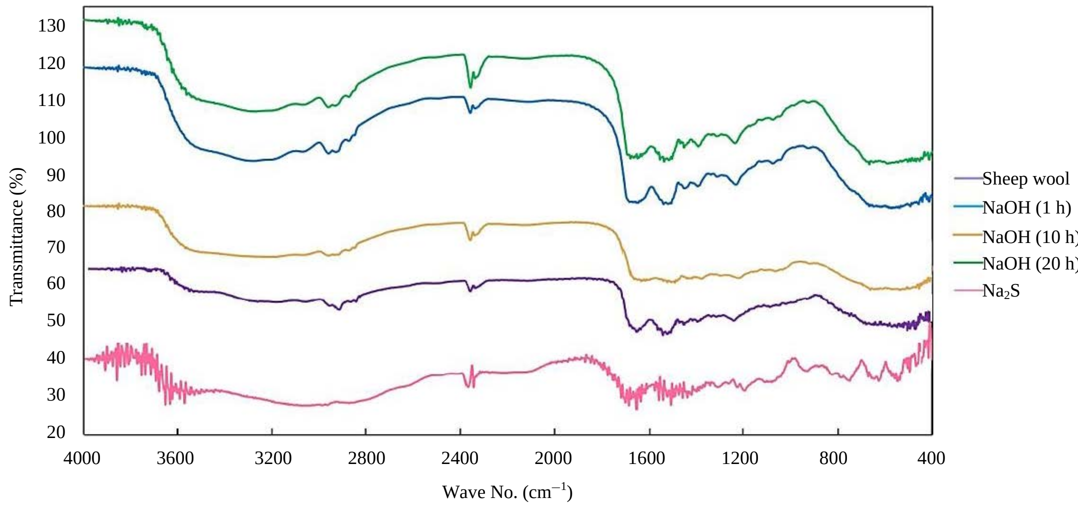

Fig. 7: FTIR analysis of alkaline treated wools

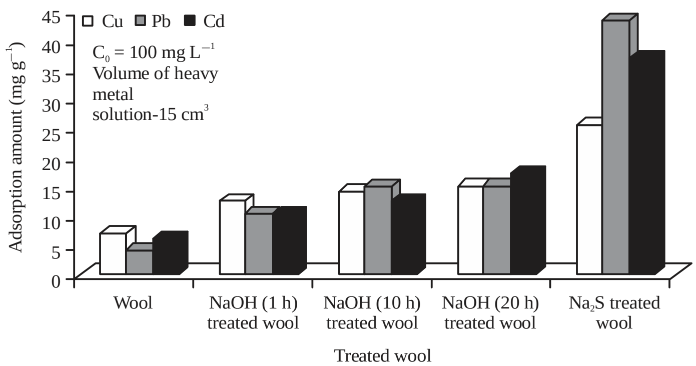

Fig. 8: Adsorption amount of the heavy metals by livestock biomasses

metals, all of the Na_2S-treated wool samples mainly comprised of C, N, O, Na, Sr and S elements. The amount of heavy metals in the surface of the Na_2S-treated wool samples were defined as 2.42% of Cu, 3.32% of Pb and 2.56% of Cd as shown in Fig. 6b, d, f.

Fourier transforms infrared spectroscopy (FTIR) measurements of the wool: The FTIR results of the sheep wool and the alkaline treated sheep wool samples were shown in Fig. 7. Untreated sheep wool and alkaline treated sheep wool had amide mode I, II and its incidental to the α-helix and β-sheet of structure of the wool. The amide I, II, III band are usually connected to vibration peak of C = O stretching; N-H bending deformation and C-H, C-C stretching; N-H, C-O bending and C-N, C-C stretching, respectively. The alkaline treated wool was compared with the untreated wool; functional groups were strongly revealed in all of the samples.

Adsorption behavior of the alkaline treated sheep wool: The heavy metal adsorption amounts of the wool and the alkaline-treated wool is shown in Fig. 8. The adsorption amount of the heavy metals was determined as 7.15 mg g^{-1} Cu in the wool, which increased to 26.04 mg g^{-1} in the Na_2S treated wool. The wool adsorbed 4.30 mg g^{-1} Pb, 6.26 mg g^{-1} Cd, which amounts increased to 43.72 mg g^{-1} Pb, 37.35 mg g^{-1} Cd in the Na_2S-treated wool samples.

Effect of treatment time of the sheep wool by NaOH solution: Heavy metal removal by NaOH treated wool was measured with different time as shown in Fig. 9. The NaOH (0.1 M) treatment was made for 1, 10, 20 h at 50°C. The initial concentrations of heavy metals were set at 100 mg L^{-1}. By increasing treatment times, the adsorption amount of heavy metal was increased[19]. The wool treated with the NaOH solution for 20 h, adsorption amount was defined as 15.19 mg g^{-1} of Cu, 15.15 mg g^{-1} of Pb and 17.32 mg g^{-1} of Cd at initial concentration of 100 mg L^{-1}.

Effect of pH: Removal effects of Cu, Pb, Cd by the alkaline treated wool on the pH effect for 3, 5, 6, 8 are shown in Fig. 10. The removal efficiency of the Cu defined as 59.91% and above, except for the Cu removal by the NaOH and the Na_2S-treated wools for pH_{eq} at 3.3-3.7. The removal efficiency was defined as 73.02% and above for the Pb solutions, 81.36% and above for the Cd removal, except for the Cd removal by the NaOH-treated wool for pH_{eq} at 3.6.

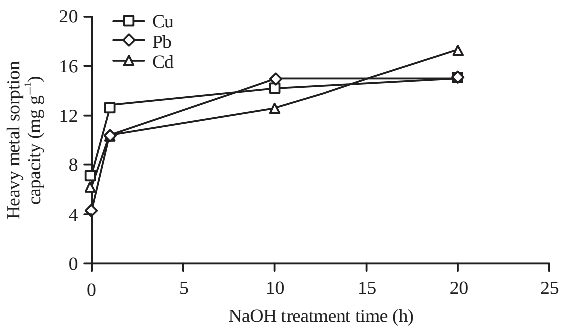

Fig. 9: Effect of treatment time of the heavy metal sorption of alkaline treated wool

Adsorption capacities and adsorption equilibrium constant of heavy metals: The experimental results of the adsorption isotherm of heavy metals were analysed using Langmuir and Freundlich isotherms. The heavy metals adsorption was analysed by Langmuir adsorption isotherms as shown in Fig. 11. The correlation coefficient (r^2) of Langmuir adsorption isotherm has defined as better than that of Freundlich adsorption isotherm and fit successfully with the heavy metals adsorption results. The heavy metals adsorption amounts were increased drastically by the treatment of NaOH and Na_2S, which was also proved by the Langmuir adsorption isotherm analysis.

DISCUSSIONS

This study was executed heavy metals adsorption using livestock biomasses of Mongolia. From result, livestock biomasses of sheep wool, sheep manure and sheep fat were ability to make adsorption of heavy metals. Especially sheep wool was adsorbed high amount of heavy metals and after alkaline treatment heavy metals adsorption amounts were increased. Mongolia is a country of animal husbandry of the nomadic and rich in sheep. The result of heavy metals adsorption proves we can to get rid of environmental problem using waste material of sheep.

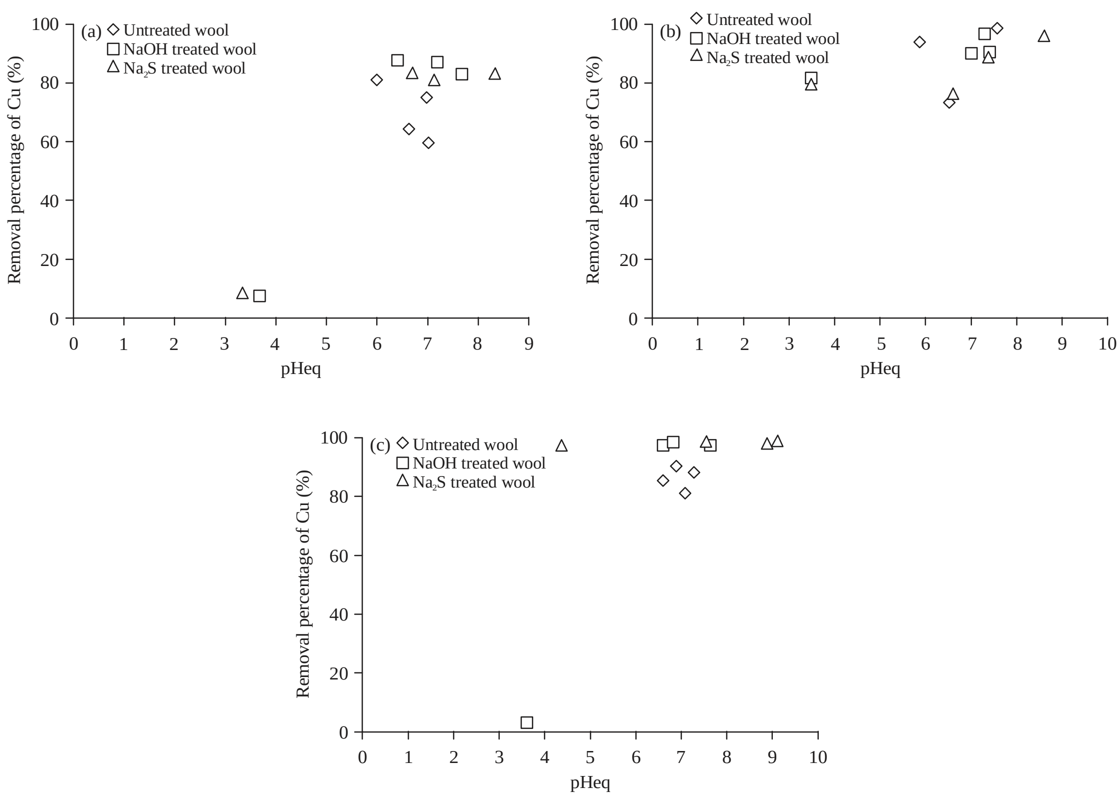

Fig. 10: Effect of pH on heavy metals removal by the untreated wool, NaOH and Na_2S treated wools (a) Cu, (b) Pb and (c) Cd

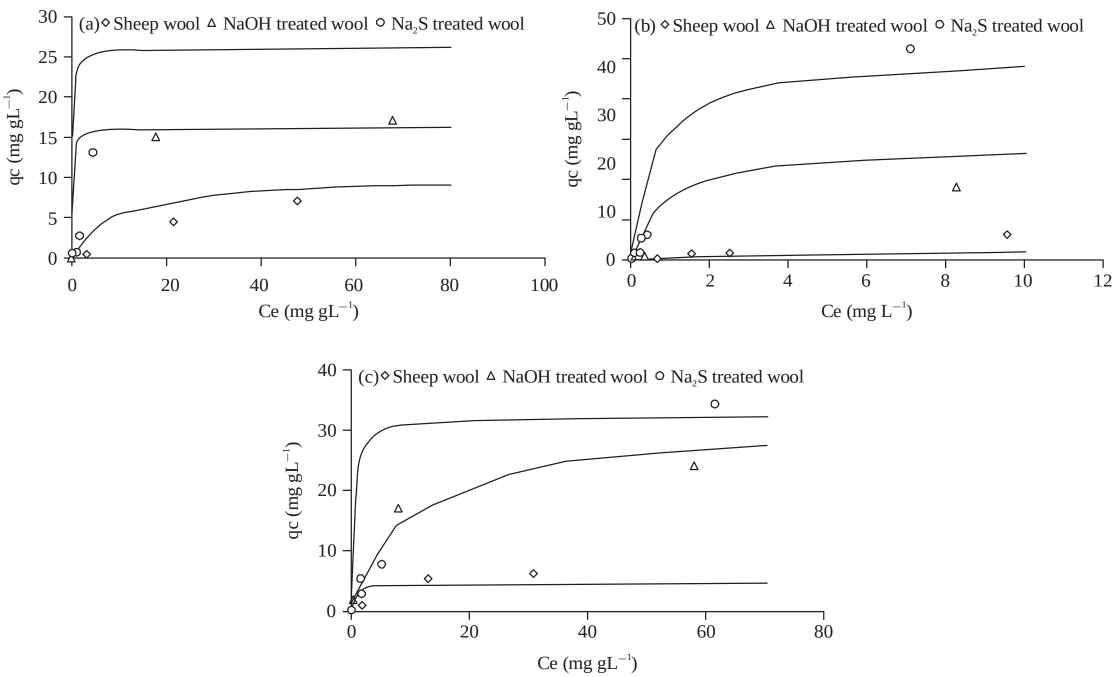

Fig.11: Adsorption isotherms of heavy metals by the untreated wool, NaOH and Na_2S treated wools (a) Cu, (b) Pb and (c) Cd

Keratin based materials used as adsorbents and removing heavy metals from waste water. The keratin based materials were used as adsorbent for removing heavy metal ions such as As^{3+} removal by wool filter[30]; Cr^{6+} removal by hybrid polyurethane membrane with 15 wt% chicken keratin[31]; Cr^{6+}, Cr^{3+}, Cu^{2+} removal by electrospun nano-fibrous membranes with 50 wt% keratin resin[32,33]; Cr^{6+} removal by chicken feather[34]; Pb^{2+} removal by poultry feather fiber[35]; and Zn^{2+} removal by chicken feather particle[36].

The adsorption capacity of heavy metals of Cu was determined 26.2 mg g^{-1} by Na_2S treated wool in this study which 61.7 mg g^{-1} of Cu by Keratin/PA6 50/50[23]; 52.2 mg g^{-1} of Cu by Duck feather treated with NaOH[21] and 29 mg g^{-1} of Cu by human hair treated with NaOH/Na_2S[25].

The results in this study similar with the study of the sheep wool treated with NaOH adsorbed 30.12 mg g^{-1} of Pb[22]; and 43.3 mg g^{-1} of Pb adsorbed by Keratin colloidal solution prepared from wool[18]; which adsorption capacity of heavy metals of Pb was determined 42.55 mg g^{-1} by Na_2S treated wool in this study.

Therefore, adsorption of Cd was determined 32.46 mg g^{-1} by Na_2S treated wool in this study that 53 mg g^{-1} of Cd by Human hair treated with NaOH/Na_2S[25].

Implications: According to the research livestock biomasses possible to use for adsorption material, adsorption capacity will increase when chemical treatment made in livestock biomasses.

Applications: This study possible to extend in range of application and treatment of sheep wool made with different kind of chemical substances. Adsorption material of sheep wool would be used for industrial level regarding capability of increase adsorption capacity.

Recommendations: Many kind of keratin based adsorption material prepared for each study which better to review characterization and application behavior of adsorption material.

Limitations: In the current study, adsorption of heavy metals by livestock biomasses determined different in each heavy metals. Adsorption material based on sheep wool is difficult to make adsorption from polluted with too many heavy metals.

CONCLUSION

This study evaluated the adsorption of heavy metals removal with the low-cost sorbents such as livestock biomasses of Mongolia. Sheep manure, sheep wool and sheep fat were defined as effective adsorbent for the heavy metals. After the alkaline treatment of the wool with NaOH and Na_2S, the adsorption amounts of the heavy metals increased effectively. The removal efficiency was determined as quite high at low initial concentration. The Na_2S and NaOH treated wool adsorbent adsorbed high amount of the heavy metals rather other adsorbents in this study. After adsorption, the biomasses adsorbents would be able to burn. The adsorbent materials of the wool treated with NaOH and Na_2S are expected to use as a filter with a sheet and a film form for the removal of the heavy metals from contaminated rivers in further study.

SIGNIFICANCE STATEMENTS

This study discovers the heavy metals adsorption ability of sheep wool that can be beneficial for removing heavy metal pollution around mining area using waste materials of livestock. This study will help the researcher to understand advantages of livestock biomasses. Furthermore, waste materials of livestock biomasses, especially sheep wool, would be efficient to remove heavy metals from mining waste.

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