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Research Article

Removal of Cobalt Metal Ion from Aqueous Solution using *Punica granatum* Stem Powder as Biosorbent

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ABSTRACT

Natural agricultural solid waste material is very cost-effective for synthesizing adsorbents utilized in the removal of heavy metal ion from economic and noneconomic wastewaters, which are numerously exploded in environmental soil and water resources. *Punica granatum* (pomegranate) stem powder (PGSP) was synthesized into nanoparticle size and practiced in the removal of cobalt metal ions. The adsorbent was synthesized in a greener manner in an aqueous medium. A batch adsorption study was carried out with parameters including the effect of pH (pH 4, 73.13%), adsorbent dose (200 mg, 70.80%), the concentration of metal ion (200 ppm, 81.56%) and effect of temperature (30°C, 71.56%). Characterization study of PGSP before cobalt metal ion adsorption and after cobalt metal ion adsorption was conducted, including scanning electron microscopy, transmission electron microscopy, X-ray diffraction analysis, fourier transform infrared spectroscopy, ultraviolet-visible spectroscopy and elemental detection analysis.

Keywords: Cobalt, Biosorbent, Characterization, Synthesis, Removal.

INTRODUCTION

Cobalt as a heavy metal has a common form of divalent and trivalent ions is increasing in soil and water texture due to the heavy waste waters as residual effluents discharged from industrial sources, including pharmaceuticals, mining, electroplating, alloy fabrication and hard metal development industries.[1,2] Excess of cobalt metal ions into the environment and their distribution in various sources of the environment, including food moieties, seriously affect human health factors.[3] Certain research researchers have stated that cobalt toxicity in body affects the muscles of heart which leads to the disease of heart muscles known as cardiomyopathy and other serious infections occurring in red blood cells such as polycythemia, as this symptom can travel towards congestive heart failure.[4,5] It is quite adequate that cobalt metal ions are essential co-factor for the growth of body tissues and is present in variety of nutritional medium relating to vitamin B12.[6] Cobalt metal ions in its divalent and trivalent state in excess can result for toxicity. The nature of cobalt suggests for its gray appearance with atomic number 27 and its atomic weight of 58.9 Da.[7] It is studied that in the environment, cobalt is naturally found in combination with naturally occurring elements such as, sulfur, copper, arsenic, magnesium and nickel. Cobalt, because of its property of ferromagnetism and higher physical constant of 1495.05℃ (melting point), and 2927℃ (boiling point), it is constantly employed in industrial sectors for the production of superalloys and hard metals, including iron, nickel and aluminum blends.[8] The most toxic cobalt metal ions are produced from the production of tungsten- carbide which is employed for strengthening the properties of heat sensitivity and alloy hardness.[9]

The ancient history of cobalt metal explains that cobaltous chloride was used in ancient medicinal therapy for the treatment of serious diseases like anemia but there were some adverse effects related with the use of cobalt in the treatment of anemia, leading to dysfunction of thyroid and causes production of goiter cells in thyroid.[10,11] The toxicity of cobalt metal ions leads to discomfort of multiple body organs related to the endocrine system, metabolic pathways, hematological functions, disorders of the cardiovascular system and effects on the nervous system.[12]

Researchers from many years has employed hundreds of methods for the removal of heavy metal ions from wastewater as deposition of various industries generating wastewater from production plants and disposed of in freshwater systems including lakes, rivers, ponds, and groundwater percolation, as this waters containing heavy metals are distributed in food chains through various pathways.[13] Adsorbent synthesis for heavy metal adsorption has been carried out by chemical, physical and green methods.[14] Chemical and physical methods include co-precipitation, sol-gel, chemical vapor, wet impregnation, electrospinning, thermal deposition, complex-directed hybridization, microwave synthesis, thermal oxidation, and hydrothermal methods.[15] Adsorbent synthesis practiced through chemical and physical methodology requires heavy use of chemicals, various instrumental techniques which leads to the generation of pollutants in the environment by any means and serious problem of deposition of pollutants.[16] The researchers largely practice adsorbent synthesized through green manner for removal of heavy metals as the starting material is plant material, including various parts, agricultural waste, animal waste, and various fungus and bacteria employed for adsorbent synthesis.[17] Adsorbent produced from environmental factors is of low cost and ecofriendly and recyclability of adsorbent can be achieved.[18]

Adsorbent synthesized from agricultural wastes includes Eucalyptus *sheathiana* bark for zinc ions, *Melaleucadio smifolia* leaf for cr^{6+} ions, tomato leaf powder for nickel ions, rice husk for zinc ions, orange peel for lead ions, pine cone for copper ions, papaya wood for copper ions, *Psidiun guvajava* leaf powder for Cd^{2+} ions, Rambai leaves for Hg^{2+}, castor seed hull for zinc ions, biomass of tobacco stem for copper, water hyacinth for Zn^{2+} and Cr^{6+}, biomass of citrus peel waste for Zr (IV) ions, modified carbon from egg sheels for Zn^{2+}, Ni^{2+} and Cd^{2+}, raw pomegranate peel for Pb^{2+} and Cu^{2+}, *Giombo persimmon* seed for Zn^{2+}, dried cactus for Cd^{2+} and Pb^{2+}, *Citrus limetta* peel for Cr(VI), *Chlorella valgaris* algae for Cd^{2+}, mango kernel for Cr(VI), bagasse pith for Zn^{2+}, jack fruit leave powder for Ni^{2+}, grape fruit peel for Cd^{2+} and Ni^{2+}, tamarind fruit peel for Ni^{2+}, cedar bark for Cu^{2+}, ulmas tree leaves for Pb^{2+}, Cu^{2+} and Cd^{2+}, coffee residue for $Pb^{2+}$and Zn^{2+}, *Tectona grandis* leaves for $Ni^{2+}$and Co^{2+}, vanda species for Pb^{2+}, mapal leaves Cd^{2+}, Cu^{2+}, Pb^{2+} and Zn^{2+}, saw dust for Cu^{2+} and Cu^{2+}, melon peel for Zn^{2+} ion removal.[19-26] Adsorbents synthesized in this green manner are composed of variety of phytochemicals embedded on the surface of adsorbent particles which largely initiates the adsorption of toxic and heavy metals.[27]

In this research pomegranate stem powder is utilized as an efficient adsorbent for the removal of cobalt metal ions through the aqueous solution. *Punica granatum* (pomegranate) stem powder is synthesized in green manner and introduced for removal of cobalt metal ions with various parameters, including pH, metal ion concentration, adsorbent dose and effect of temperature. Stem adsorbent was subjected to analytical instrumentation before and after cobalt metal ion adsorption.

MATERIAL AND METHODS

P. granatum Stem Adsorbent Preparation

P. granatum stem were collected from an organic pomegranate farm in Kolhar village, Rahata Taluka, Maharashtra, India. Collected stem material was mascaraed into small pieces, which was washed with distilled water to get rid of foreign particles and subjected for drying in direct sunlight for fifteen days. Further stem material was grinded into very fine powder and meshed with cotton cloth to obtain very nano size particles. This fine light brown color powder was dried in oven at 90°C for 10 hours, resulting in a dark black-brown color. This obtained powder is stored in container, which is used for biosorption studies. No chemical was employed in adsorbent preparation.

Batch adsorption studies

Removal of cobalt ions was studied through various parameters employed by *P. granatum* stem powder (PGSP). The chemicals used were of AR grade for parametric study with purity of high range. Adsorbate stock solution contained 1000 mg/L of cobalt ions prepared by dissolving cobaltous chloride with accurate measurement in double distilled water.[28] The stock solution for investigation was diluted for the initial working solution. Adsorption experiments was carried out with 50 mL of cobaltous ion solution of desired concentration with the addition of 200 mg *P. granatum* stem powder (PGSP) in a stoppered flask of 250 mL, as an experiment of adsorption was managed at 30°C which is near room temperature, further stirring the mixture combination for 60 minutes at pH 3 was altered with solutions of 1N NaOH and 1N HCl.[29,30] Cobaltous ion solution was removed from the stirrer in a proper time of 15 minutes and separation of adsorbent was practiced with Whatman filter paper No.41. Cobalt ion samples were examined on UV-visible spectrophotometer for the study of parameters including pH, adsorbent dose, and metal ion concentration and effect of temperature[31-34] Reaction time for this study is 240 minutes with 15 minute of time interval. The formula derived the percentage of removal of cobalt metal ions from absorbance,

$$\%\textbf{removal}= A_0\text{-}A_t \ / \ A_0$$

Where, A_0= Absorbance at initial time
A_t= Absorbance at final time

RESULTS AND DISCUSSION

Effect of pH cobalt ion adsorption

Various pH study verified the pH parameter (2, 4, 6, 8, 10, 12). Cobalt ion solution's initial concentration is 150 ppm, PGSP dose 200 mg, 30°C temperature, cobalt ion solution 50 ml, 360 rpm agitation speed. Results indicated in Fig. 1 and Table 1 show 73.13% removal of cobalt ions at maximum pH 4.[35,36] It is observed that cobalt ion removal increases from pH 2 to 4 (47.05–73.13%) and again decreases from pH 6 to 12 (63.21, 59.52, 57.74, 56.41). It is narrated that the adsorbent surface is furnished with enamourous functional groups containing organic molecules and phytoconstituents which achieves the maximum removal of cobalt ions. Cobalt ions removal efficiency is influenced by hydrogen ion concentration in aqueous solutions which affects removal capacity of cobalt metal ions, so at an acidic pH of 4, hydrogen ion concentration is high on the surface if adsorbent and gradually decreases as pH increases.[37] Hydrogen ion concentration tends to replace positive ions present on active sites, which affects biomass degree of ionization during metal ion removal.[38]

Effect of PGSP dose on cobalt ion adsorption

Cobalt ion adsorption by PGSP was experimented with through PGSP dose (50, 100, 150, 200, 250 mg), cobalt ion initial metal ion concentration 150 ppm, temperature of 30°C, reaction time of 240 minutes, cobalt ion solution of 50 mL, agitation speed of 360 rpm and reaction carried at pH 4.[39] Maximum cobalt ion removal is observed at 200 mg adsorbent dose (70.18%) and removal decreases as the adsorbent dose increases, as shown in Fig. 2 and Table 2. The constant nature of cobalt ion removal on the increase of the adsorbent dose is an increase in the saturation of active adsorbent sites which becomes unavailable for the adsorption of cobalt metal ions on the surface of the biosorbent.[40]

Cobalt Metal ion Concentration

Metal ion concentration study was carried at various concentrations of cobalt metal ion solution (50, 100, 150, 200, 250, 300 ppm), temperature 30℃, agitation speed 360 rpm, PGSP 200 mg and reaction carried at pH 4 with a reaction time of 240 minutes. High removal of metal ion was achieved at 250 ppm (82.56) as shown in Fig. 3 and Table 3. The experimental study elaborates that as metal ion concentration increases from 50 ppm to 250 ppm, the metal ion removal capacity of PGSP increases (68.59–82.56%). It is observed that metal ion concentration does not affect the adsorption capacity of biosorbent because maximum adsorption active sites are available on the biosorbent surface as there is no presence of saturation of metal ion solution.[41]

Effect of Temperature

Effect of temperature (10, 20, 30 and 40°C), adsorbent 200 mg, pH 4, agitation speed 360 rpm, metal ion concentration 150 ppm and reaction time was 240 minutes. Temperature condition was maintained through hot plate sensitization, as the actual temperature of the reaction mixture was attained thought the experiment. Maximum metal ion removal was attained at 30°C (71.56%), show in Fig. 4, Table 4. It is observed that at lower temperature metal ion removal efficiency is less but as temperature increase metal ion removal increases and remains constant further as temperature increases.[42]

CHARACTERIZATION STUDY

P. granatum stem powder was subjected to analytical characterization before cobalt metal ions adsorption and after cobalt metal ions adsorption, as the characterization study explained that the PGSP is capable of adsorbing cobalt ions efficiently, which is explained through scanning electron microscopy (SEM), transmission electron microscopy (TEM), X-ray diffraction analysis (XRD), Elemental detection analysis (EDS), Fourier transforms infrared spectroscopy (FTIR), Ultraviolet visible spectroscopy (UV-VIS).

Table 1: Effect of pH

pH	2	4	6	8	10	12
%removal	47.05	73.13	63.21	59.52	57.74	56.41

Table 2: Adsorbent dose

PGSP dose (mg)	50	100	150	200	250	300
%removal	33.92	45.56	60.28	70.18	70.14	68.53

Table 3: Metal ion concentration

Metal ion conc. (ppm)	50	100	150	200	250
%removal	68.59	73.25	78.63	81.56	82.56

Table 4. Effect of temperature

Temperature (℃)	10	20	30	40
% removal	25.36	37.89	71.56	68.32

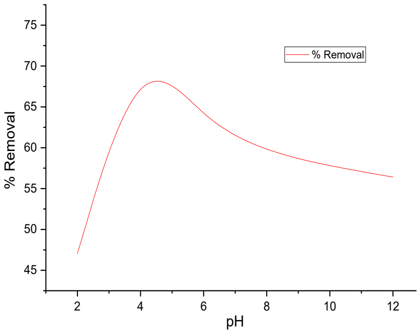

Fig. 1: Effect of pH

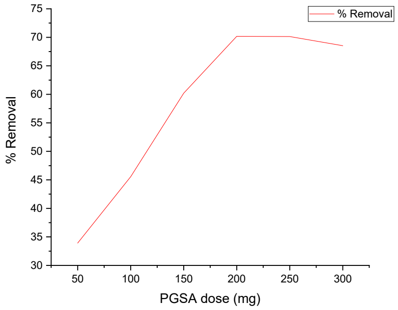

Fig. 2: Adsorbent dose

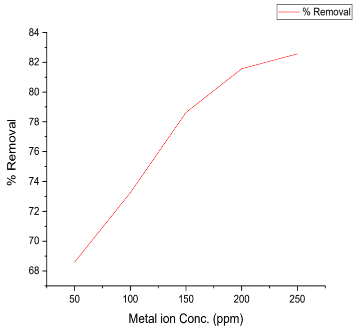

Fig. 3: Metal ion concentration

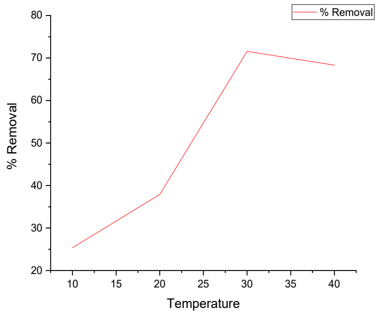

Fig. 4: Effect of temperature

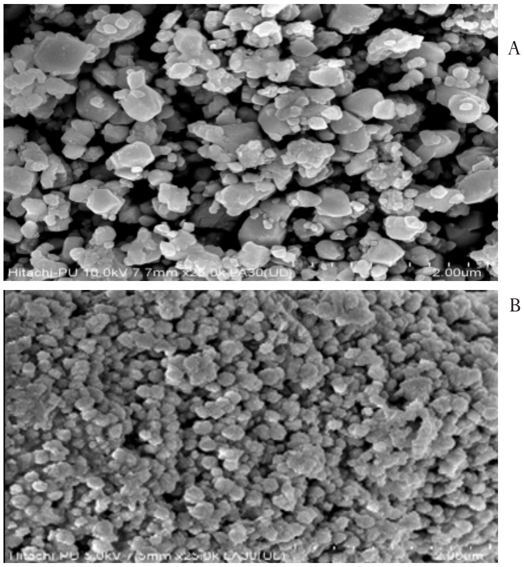

Fig. 4: (A) SEM (before adsorption) (B) SEM (after adsorption)

SEM Analysis

Scanning electron microscopy of PGSP before and after adsorption of metal ions was practiced, shown in Figs 4. A and B. Before the adsorption of cobalt metal SEM analysis shows the square cubic nature of nanoforms with nanosize exhibiting of 20 to 30 nm and after adsorption of cobalt metal ions changes occurs has been observed in SEM analysis with spherical and oval shape and particle size of 12 to 25 nm.[43] This difference in SEM analysis results in cobalt metal ion adsorption has been occurred.

TEM analysis

Transmission electron microscopy of adsorbent powder before and after cobalt ion adsorption was carried out and show in Figs 5A and B. Adsorbent powder before adsorption shows small nano flowers appearance and after cobalt ion adsorption square and hexagonal nature of particles appears, so this difference in TEM analysis confirms cobalt ion adsorption by PGSP.

XRD Analysis

X-ray diffraction analysis of PGSP was examined, as PGSP before cobalt metal ion adsorption shows a crystalline nature with a particle size of 9 nm with miller indices (110, 111, 200, 211, 220, 311, 222). XRD for PGSP after cobalt metal ion adsorption resulted for hexagonal Wurtize nature of particles with size of 6 nm and miller planes (100, 002, 101, 102, 110, 103, 200, 112, 201).[44] The difference in XRD nature is observed in the analysis, proving the high adsorption of cobalt ions, show in Figs 6A and B.

FTIR Analysis

P. granatum stem powder before and after the adsorption of cobalt metal ions was subjected to Fourier transform infrared spectroscopy analysis. PGSP consists of an enamourous presence of organic, inorganic, phytochemicals and phytoconstituents. PGSP before cobalt metal ion adsorption shows FTIR data as, hydroxyl (OH) at 3758.24, 3928.64, 3552.46 cm^{-1} for Ar-OH, 3414.11 cm^{-1} determines -OH group, 3236.01 cm^{-1} N-H stretch, alkene -CH show 2920.05 and 28.53.29 cm^{-1}, = CO show 2563.67 cm^{-1}, silicon presence at 2034.18 cm^{-1}, ketonic group at 1740.81 cm^{-1}, (–C=C-) at 1617.85 cm^{-1}and 1638.81 cm^{-1}, amines (N-H) peak 1583.80, 1507.26 cm^{-1} show aromatic (-C-C-) group, 1432.58 cm^{-1} phytoconstituents (-C=O) carbonyl group, 1406.49 cm^{-1} show (–C-O-H-) stretching, 1384.41 cm^{-1} for alkyl (-CH$_3$), –CH-CH$_3$- group at 1330, 1257.33 cm^{-1} for alkyl halides (-CH-) with wagging stretch, –C-O- of alkyl ester at 1147.68, 1232.32 cm^{-1} and 1050.95, 851.56 cm^{-1} alkenes (=C-H) out of plane out of plane, 883.20 cm^{-1} for various active sites –C-OH, -C-O-C- and –CH, 822.22 cm^{-1} resulting for carbohydrate.[45] PGSP after cobalt metal adsorption shows variant frequency bands of 479.61 and 619.49 cm^{-1} for the presence of cobalt ion peak associated with the complex formation with phytochemicals and hence proved that PGSP has an efficient capacity to adsorb cobalt metal ions. show in Figs 7A and B.

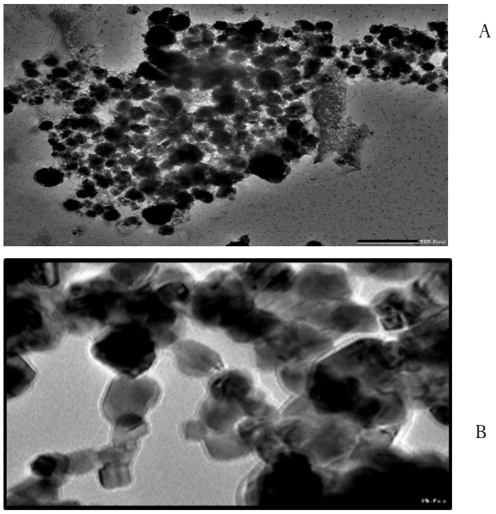

Fig. 5: (A) TEM (before adsorption) (B) TEM (after adsorption)

UV-Visible Spectroscopy

PGSP before adsorption dissolved in an aqueous medium, adsorbent solution filtered and analyzed with UV-visible shows maximum

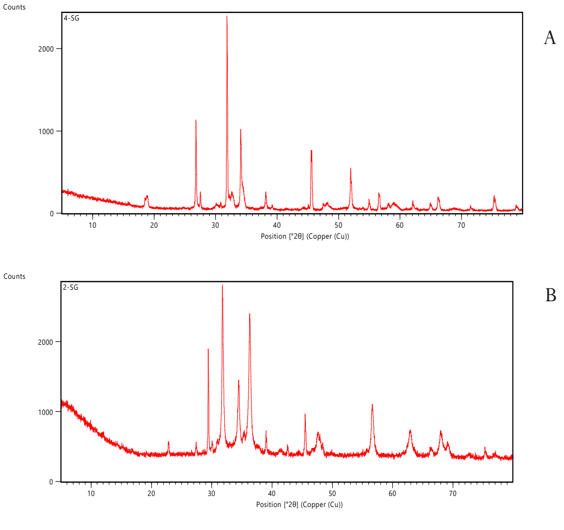

Fig. 6: (A) XRD (before adsorption) (B) XRD (after adsorption)

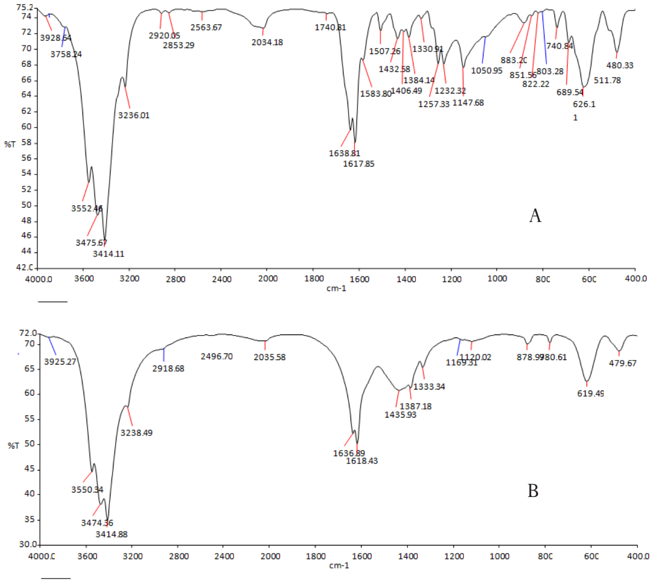

Fig. 7: (A) FTIR (before adsorption) (B) FTIR (after adsorption)

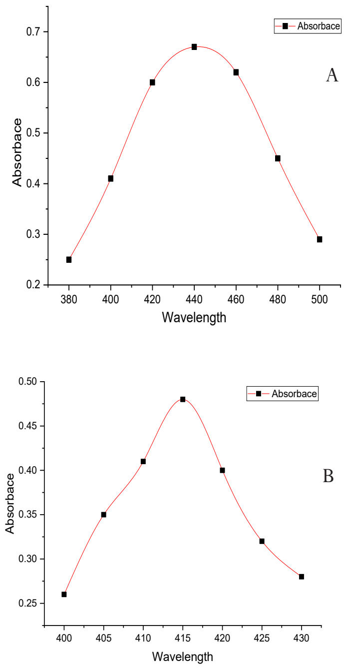

Fig. 8: (A) UV-Visible (before adsorption) (B) UV Visible (after adsorption)

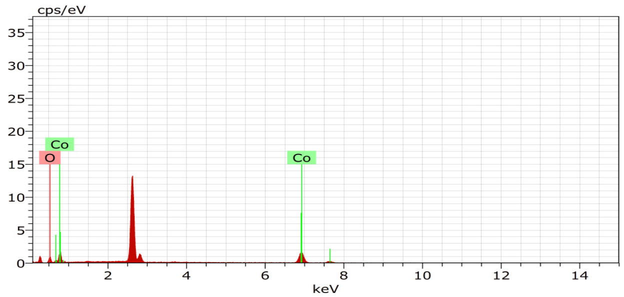

Fig. 9: EDS analysis (after adsorption)

absorbance at 440 nm and PGSP with adsorbed cobalt metal ions shows maximum absorbance at 415 nm, show in Figs 8A and B.

EDS Analysis

Elemental detection analysis of PGSP after cobalt metal ion adsorption shows the presence of cobalt, oxygen and hydrogen as cobalt (43%), oxygen (45%) and hydrogen (23%), show in Fig. 9. EDS analysis confirms that *P. granatum* stem powder is capable of adsorbing cobalt metal ions.

CONCLUSION

P. granatum stem powder (PGSP) as low-cost adsorbent was synthesized for the removal of cobalt metal ions. Synthesis was carried out in an aqueous medium as the synthesized adsorbent was subjected to various characterizations before cobalt metal ion adsorption and after cobalt metal ion adsorption. Characterization includes scanning electron microscopy, transmission electron microscopy, X-ray diffraction analysis, fourier transform infrared spectroscopy, Ultraviolet-visible spectroscopy and elemental detection analysis. Parameters studied under adsorption as effect of pH, adsorbent dose, metal ion concentration and effect of temperature. It is concluded that *P. granatum* stem powder can efficiently be employed to remove cobalt ions from wastewaters.

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CONFLICT OF INTEREST

None.

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