Experimental Procedure

3.0 EXPERIMENTAL PROCEDURE

Excessive release of heavy metals into the environment due to industrialization and urbanization has posed a great problem worldwide. The presence of heavy metal ions is a major concern due to their toxicity to living organisms. The removal of metal ions is necessary because of their harmful effect to aquatic life. The adsorption process is being widely used for the removal of heavy metals from waste streams. A number of research works are done using different agro waste adsorbents in their activated carbon form. To remove the pollutants, a novel adsorbent, human hair was tried in the present study.

The experimental procedure pertaining to the study entitled "Adsorption of nickel(II) and chromium(VI) from synthetic metal solutions using human hair as adsorbent" is discussed under the following headings.

3.1 COLLECTION OF HUMAN HAIR
3.2 CHARACTERISATION OF HUMAN HAIR
3.3 PREPARATION OF SYNTHETIC METAL SOLUTION
3.4 TREATMENT OF THE SYNTHETIC METAL SOLUTIONS WITH HUMAN HAIR AS AN ADSORBENT
3.5 STUDY OF THE KINETICS OF NICKEL AND CHROMIUM ADSORPTION ON HUMAN HAIR
3.6 STATISTICAL ANALYSIS

3.1 COLLECTION OF HUMAN HAIR

Human hair was collected from Avinashilingam University hostel students, beauty parlour and barber shop from both male and female subjects. Small polythene covers were distributed. In hostel, girls were requested to collect the hair samples which dropped while combing their hair. In beauty parlour and barber shop, hairs were collected during hair cutting. The collected hair samples

were washed several times with tap water and rinsed with alcohol. The samples were dried in an oven at 75±5°C and then it was cut into tiny bits for the preparation of powdered human hair used in sorption tests (Plates 1 and 2).

PLATE 1
HUMAN HAIR

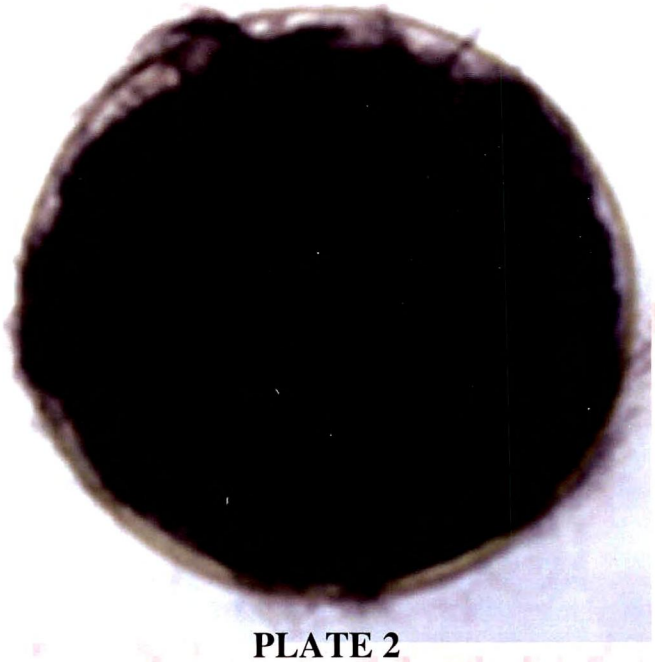

PLATE 2
POWDERED HUMAN HAIR

3.2 CHARACTERISATION OF HUMAN HAIR

The powdered hair samples were analysed for their ash content, moisture content, bulk density, water-soluble matter, acid soluble matter, and pH. All the estimations were carried out in three replicates.

3.2.1 Ash content (Raghuramulu *et al.*, 1983)

An empty crucible was cleaned well and heated to redness over a Bunsen burner, cooled in a desiccator and weighed. Heating, cooling and weighing were repeated to obtain constant weight.

2.0g of the powdered hair sample was taken in the pre-weighed crucible and the total weight was noted. Then the crucible was heated over a Bunsen burner until the sooty flame disappeared. Then kept the crucible in the muffle furnace at 550°C for 6 hours. The crucible was then cooled in a desiccator and weighed. (Weight of the crucible + Ash) – (Weight of the empty crucible) gives the weight of the ash content of 2.0g of the material. From this, the percentage of ash content was calculated.

3.2.2 Moisture content (Raghuramulu *et al.*, 1983)

An empty silica crucible with lid was cleaned well and heated to redness over a Bunsen burner. Cooled in a desiccator and weighed. This procedure was repeated until a constant weight was obtained. The difference in the weight of the crucible with the powdered hair sample before and after heating gives the moisture content of the material. From this value the percentage of moisture content was calculated.

3.2.3 Bulk density (Pearson, 1970)

Placed 20g of dried powdered hair sample in a 100 ml measuring cylinder. Levelled off the powder up to the mark and measured the bulk density in g/ml.

3.2.4 Matter soluble in water (Pearson, 1970)

Added 250 ml of water to 2.5g sample in a 600 ml beaker. After allowing to stand for 5 minutes, poured through a dried and weighed filter paper. After washing the filter well with water, dried and weighed the insoluble matter and calculated the solubility figure.

3.2.5 Matter soluble in acid (Pearson, 1970)

Added 100 ml of concentrated hydrochloric acid to 1.0g sample in a 250 ml of a beaker. After allowing to stand for 5 minutes, poured through a dried and weighed filter paper. After washing the filter well with water, dried and weighed the insoluble matter and calculated the solubility figure.

3.2.6 pH (ISI, 1984)

Weighed accurately 5g of the sample and added 50 ml of distilled water. Allowed the contents to soak for 1 hour. Stirred the contents for 10 to 15 minutes, preferably with the help of a mechanical stirrer to obtain a uniform aqueous suspension. Recorded the pH of the suspension in a pH meter.

3.3 PREPARATION OF SYNTHETIC METAL SOLUTION

Synthetic nickel(II) and chromium(VI) solutions were prepared by dissolving nickel chloride in distilled water, to have 0.2mg of nickel/ml and by dissolving potassium dichromate in distilled water, to have 0.2mg of chromium/ml and binary metal solution was prepared by dissolving nickel chloride and potassium dichromate in distilled water in order to have 0.1mg of nickel and 0.1mg of chromium/ml.

3.4 TREATMENT OF THE SYNTHETIC METAL SOLUTIONS WITH HUMAN HAIR AS AN ADSORBENT

The metal adsorption studies were carried out by batch mode using different pH, temperature, adsorbent dose, initial concentration of metal ions and contact time, in order to determine the optimum experimental condition.

Batch adsorption experiments (Singh and Lal, 1992)

Batch adsorption studies were carried out using glass stoppered conical flasks separately containing 50 ml of water and 50 ml of synthetic nickel solution, 50 ml of chromium solution and 50 ml of binary salt solutions with the adsorbent in each. Batch adsorption experiments were carried out under five different experimental conditions namely different pH (pH 4, 5, 6, 7 and 8), different temperature (20°C, 25°C, 30°C, 35°C and 40°C), different adsorbent dose (1, 2, 3, 4 and 5g), different dilutions (1:1, 1:2, 1:3, 2:1 and 3:1) and different contact time (15, 30, 45, 60 and 75 min).

The flasks were shaken in an electric shaker. After equilibrium period, the contents of the flask were filtered using Whatman No. 4 filter paper and the concentrations of nickel and chromium in the solutions were determined using photometer 105.

3.4.1 Estimation of Nickel(II) by Spectroquant test kit method

Principle

Nickel(II) ions were oxidized by iodine and then were transferred with dimethylglyoxime in an ammonia solution to get a red-brown colour complex. The colour developed is determined photometrically.

Reagents

1 bottle of reagent Ni-1 (iodine)

1 bottle of reagent Ni-2 (dimethylglyoxime)

1 bottle of reagent Ni-3 (ammonia)

Procedure

Pipetted out 5 ml of the experimental solutions into a test tube. Then added one drop of reagent Ni-1 and mixed well. A slight yellow colouration appeared in each tube. Left to stand for one minute. Added 2 drops of reagent Ni-2 and Ni-3 in each tube and mixed well. Left to stand for two minutes. The reddish brown colour formation was read against a blank in the photometer.

3.4.2 Estimation of Chromium(VI) by Spectroquant test kit method

Principle

In weakly phosphoric solution chromium(VI) ions react with diphenyl carbazide to form chromium(III) and diphenyl carbazone, which form a red-violet complex. This complex is determined photometrically.

Reagents

1 bottle of reagent Cr-1 (diphenylcarbazide)

1 bottle of reagent Cr-2 (phosphoric solution)

Procedure

One microspoonful (in the cap of the Cr-1 bottle) of reagent Cr-1 was placed into a dry clean test tube. To this added 6 drops of reagent Cr-2 and shaken vigorously until the reagent Cr-1 was completely dissolved. Then pipetted out 5 ml of the experimental solutions into each tube and mixed well. Left to stand for one minute. A reddish-violet colour was developed. Then samples were filled into the cuvette and the colour development was measured against blank in the photometer.

3.5 STUDY OF THE KINETICS OF NICKEL AND CHROMIUM ADSORPTION ON HUMAN HAIR

Nickel and chromium concentrations after adsorption at varying experimental conditions were measured by photometer 105. The results obtained with the adsorption of nickel(II) and chromium(VI) on human hair under varying experimental conditions was studied using the adsorption isotherm. The study of adsorption isotherm in any adsorption process is helpful in determining the adsorption capacity of the selected material under varying experimental conditions and thus helps in selecting the adsorbent for the removal of any metal ion.

The well known classical Freundlich adsorption isotherm represents the relationship between the amount of the metal absorbed per unit mass of the adsorbent and equilibrium concentration of the metal, which has been attempted in the present study.

The Freundlich adsorption isotherm is given as follows (Ramu *et al.,* 1992)

$$x/m = k_f Ce^{1/n}$$

The linearised form of equation is

$$\log x/m = \log k_f + 1/n \log Ce$$

where,

x/m is the amount of the metal sorbed per unit weight of sorbent (mg/g)

k_f is the measure of sorption capacity

1/n is the measure of sorption intensity

Ce is the equilibrium concentration of the residual metal ions in solution

The Langmuir isotherm model is given as follows (Al-Asheh *et al.,* 2002)

$$Q = q_{max} bCe + q_{max}$$

The linearised form is

$$1/q = 1/q_{max} bCe + 1/q_{max}$$

where,

q is the amount of metal sorbed per unit dry weight of sorbent at equilibrium (mg/g)

Ce is the residual metal ion concentration left in solution after binding

q_{max} is the maximum possible amount of metallic ion sorbed per unit weight of sorbent and

b is the equilibrium constant related to the affinity of the binding sites for the metal ions.

3.5 STATISTICAL ANALYSIS

The level of nickel and chromium adsorbed on human hair at different conditions namely pH, temperature, adsorbent dose, initial concentration of the metals and contact time were compared by performing one way analysis of variance.