

*EXPERIMENTAL
PROCEDURE*

3.0 EXPERIMENTAL PROCEDURE

Heavy metal contamination is a main source of water pollution. It is well known that some metals can be poisonous or otherwise toxic to human beings. Copper is a widely used metal. There are many potential sources of copper pollution. A number of techniques such as chemical precipitation, solvent extraction, electrodialysis, evaporation, membrane separation and activated carbon have been employed to remove heavy metals from wastewater. In recent years, the need for safe and economical methods for the elimination of heavy metals from contaminated waters has necessitated research interest towards the production of low cost alternatives to commercially available activated carbon. In the present study agrowastes such as powdered orange peel, rice husk and sugar cane bagasse were selected as adsorbents because of their high availability

The experimental procedure pertaining to the study entitled “Adsorption of copper from synthetic metal solutions using selected agrowastes” is discussed under the following headings:

- 3.1 Collection and preparation of powdered agrowastes (orange peel, rice husk and sugar cane bagasse)
- 3.2 Characterization of the powdered agrowastes
 - 3.2.1 Ash content (Raghuramulu *et al.*, 1983)
 - 3.2.2 Moisture content (Raghuramulu *et al.*, 1983)
 - 3.2.3 Bulk density (Pearson, 1970)
 - 3.2.4 Matter soluble in water (Pearson, 1970)
 - 3.2.5 Matter soluble in acid (Pearson, 1970)

3.3 Preparation of synthetic metal solution

3.4 Treatment of the synthetic metal solutions with the selected the powdered agrowastes as adsorbents

3.5 Estimation of copper by Kit method

3.6 Study of the kinetics of copper adsorption on the selected powdered agrowastes

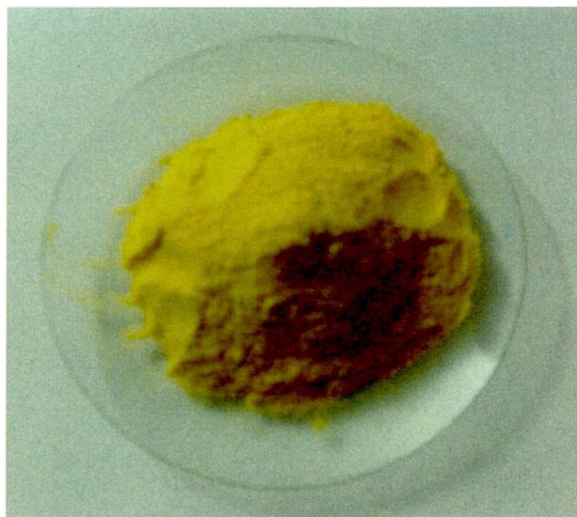
3.1 Collection and preparation of powdered agrowastes (orange peel, rice husk and sugar cane bagasse)

Orange peel, sugar cane bagasse collected from juice shops and rice husk collected from rice mill were dried in sunlight to ensure complete dryness. The dried materials were ground and then used in the sorption tests.

**PLATE 1
ORANGE PEEL**



**PLATE 2
ORANGE PEEL POWDER**



**PLATE 3
RICE HUSK**



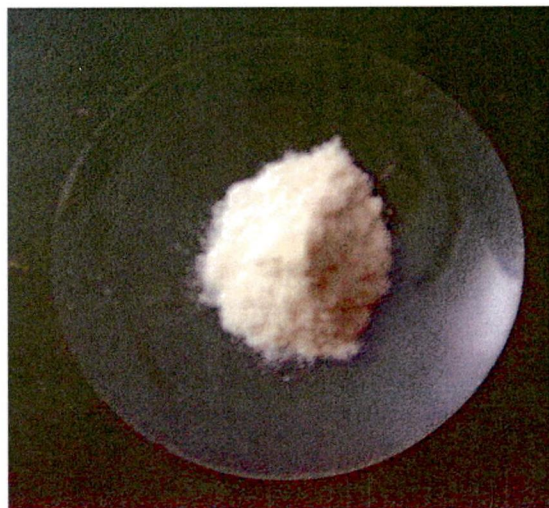
**PLATE 4
RICE HUSK POWDER**



**PLATE 5
SUGARCANE WASTE**



**PLATE 6
SUGARCANE BAGASSE POWDER**



3.2 Characterization of the powdered agrowaste samples

The powdered agrowaste samples were analyzed for their ash content, moisture content, bulk density, water soluble matter and acid soluble matter. 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 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. The percentage of ash content was calculated as

$$\text{Ash content (\%)} = \frac{\text{weight of the ash}}{\text{weight of the sample taken}} \times 100$$

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 agrowaste samples before and after heating gives the moisture content of the material. From this value the percentage of moisture content was calculated as

$$\text{Moisture content (\%)} = \frac{\text{Initial weight} - \text{Final weight}}{\text{weight of the sample}} \times 100.$$

3.2.3 Bulk density (Pearson, 1970)

Placed 20g of dried powdered 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 paper 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 beaker. After allowing to stand for 5 minutes, poured through a dried and weighed filter paper. After washing the filter paper well with water, dried and weighed the insoluble matter and calculated the solubility figure.

3.3 Preparation of synthetic metal solution

Synthetic copper solution was prepared by dissolving 0.393g of cuprous sulphate. $5H_2O$ in 100ml of distilled water, so as to have 1mg of copper/ml.

3.4 Treatment of the synthetic metal solutions with the selected powdered agrowastes as adsorbents

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 using the powdered agrowastes (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 copper solution with the adsorbents. The experiments were carried out under five different experimental conditions namely different pH (pH 2,3,4 and 5), different temperature (20°C, 25°C, 30°C, 35°C and 40°C), different adsorbent dose (0.5, 1, 1.5, 2 and 2.5g), different contact time (15, 30, 45, 60 and 75 min) and different initial concentrations of copper (20mg/100ml, 40mg/100ml, 60mg/100ml, 80mg/100ml and 100mg/100ml).

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 copper in the solutions were determined using Photometer 105.

3.5 Estimation of copper by Kit method

In an ammoniacal medium, copper II ions react with cuprizone to form a blue complex that is determined photometrically.

Reagents

Cu 1 reagent

Cu 2 reagent

Procedure

Pretreated sample (10-30°C)	5.0ml	Pipetted into a test tube
Reagent Cu-1	1 level green dosing spoon	Added and shook vigorously until the reagent was completely dissolved
Reagent Cu-2	5 drops	Added and mixed

3.6 Study of the kinetics of copper adsorption on the selected powdered agrowastes

Copper concentrations after adsorption at varying experimental conditions were measured in Photometer 105. The results obtained with the adsorption of copper on orange peel, rice husk and sugar cane bagasse under varying experimental conditions were 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 adsorbed per unit mass of the adsorbent and the 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):

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

The linearised form of equation is

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

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

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

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

$$Q = q_{\max} b C_e + q_{\max}$$

The linearised form is

$$1/q = 1/q_{\max} b C_e + 1/q_{\max}$$

where,

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

C_e 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.