SUMMARY

The work recorded in this thesis afford possibilities in techniques of waste immobilization utilizing activated carbon (CS-55) prepared from locally, available, agro-residue (Rice Husk) by one-step steam activation procedure in N_2–flow using fluidized-bed reactor as well as an opportunity to study the adsorption process. It is divided into three chapters:

Chapter 1:

An introduction describes a literature survey on the sources of contamination of water pollution and the various methods of waste water treatment including adsorption process in details. The survey includes causes and types of adsorption, adsorption isotherms, factors influencing adsorption of organic and inorganic analytes (e.g. equilibrium time, sorption rate, carbon mass, pH, initial concentration.), contacting systems and modes of operation. A survey on activated carbon is involved to describe: raw materials suitable for production of activated carbon; manufacture of active carbon and the surface chemistry of activated carbon .

Chapter 2.

Includes the procedure for preparation of activated carbon (CS-55) from Rice Husk using fluidized bed reactor. Also, the physical, chemical and surface techniques used for characterization of prepared carbon (CS-55) are given. In this concern, the surface area of the carbon (CS-55) calculated from N_2- gas adsorption during the BET equation is described . In order to gain better insight on the structure of the carbon (CS-55)

infrared spectrometry and scanning electron micrograph have been studied as well as the elemental analysis was done.

The potentiality of the carbon (CS-55) for adsorption of some inorganic and organic contaminants in batch mode was investigated. In this concern, some selected heavy metals, namely, uranium, copper, zinc was investigated using UV- spectrophotometer. Also, UV-spectrophotometer is directed to evaluated the sorption behavior of phenol- as an organic model- by the carbon (CS-55). The factors affecting the sorption process (e.g. equilibrium time, sorption rate, adsorbent mass, pH, and isotherm) were carried out for all the contaminants under investigations.

The magnitude of the interactions has been made in batch mode with 3- binary systems composed of metal ion with phenol compound whose individual adsorption properties have been studied on single basis. The 3- binary systems [(i) phenol + UO_2^{2+} ; (ii) phenol + Cu^{2+} ; and (iii) phenol +Zn^{2+}] were analyzed using UV- spectrophotometer without any interference.

Finally, the column performance parameters for phenol compound using carbon (CS-55) are evaluated. In this concern, several parameters: (e.g. substituent group, initial solute concentration, solution flow rate and bed depth) were studied . The Bed Depth Service Time (BDST) model was applied on phenol column data. Also, the evaluation of the carbon (CS-55) for removing phenol and uranium in one-process (i.e. binary- basis) has been made .

Chapter 3.

It describes the results and discussion of the experimental work . This chapter is divided into 3 parts as follows:

The first part, characterization of the carbon (CS-55), describes the physicochemical nature of the prepared carbon (CS-55) used in this study. In this concern, it was found that the carbon (CS-55) is a fine particulate matter (particle size 1-1.25 mm). The apparent density is 0.1 g / cm ^{3} and the packed density is 0.25 g / cm ^{3} with pH = 9.12 i.e. it is H-type carbon and this means that a positive surface charge over wide pH-rang due to the N_2 inert gas used during pyrolysis and cooling to low temperature in a similar environment . i. e., the carbon is exposed to oxygen only at low temperature and the oxidizing conditions necessary for acidic functional groups to form are minimized. The principal constituents of (CS-55) are: 18.2 % C ; 1.3 % H ; 18.4 % O and 62.11 % ash. The (C/H) quotient-the aromaticity of the carbon is 14 for (CS-55). This proves that the prepared carbon is suitable for treatment of organic and inorganic analytes due to (i) its high ash constituents that contain excess (Si- OH) groups which are responsible for simple ion- exchange with metal ion. (ii) the carbon content and the surface area which responsible for organic adsorption. The BET surface area of (CS- 55) is 90 m^2 / g and the scanning electron microscope (SEM) shows the pores are nearly spherical and their internal diameter < 2 nm. (i.e. micropore). The FTIR shows the surface hydroxylic, siloxane and carboxylic groups. Some aromaticity is assigned by symmetrical and asymmetrical stretching- CH_2 groups.

In the second part of the results and discussion, the sorption behavior of some selected metal ions (UO_2^{2+} , Cu^{2+} and Zn^{2+}) and phenol are studied in batch mode using carbon (CS- 55).

The first section of this part is devoted to the uptake of these pollutants by carbon (CS-55) on single solute basis using UV-spectrometry for analysis. In this concern, the equilibrium takes place within 3-4 h for metal ions (i.e. fast kinetic) compared to equilibrium for phenol (equilibrium time takes place after 48 h i.e. slow kinetic). All the adsorption systems obeyed the Lagergren first order kinetic model with rate average constant ($K_{ad} \simeq 0.023$ min^{-1}) for metal ions under investigations and for phenol ($K_{ad} = 0.052$ h^{-1}). The intraparticle diffusion rate constants for the different metal ions represented by K_p follows the following order: $UO_2^{2+} > Zn^{2+} > Cu^{2+}$ which is similar to the ionic radii order of the same metal ions. It was found that the adsorption isotherms in single component systems for all the selected metals ions (UO_2^{2+} , Cu^{2+} and Zn^{2+}) fit the two- parameter isotherm models (Langmuir and Freundlish). Accordingly, the adsorption uptake follows the: $UO_2^{2+} > Cu^{2+} > Zn^{2+}$ and in general, the lower the pH at which cationic metals form aqueous hydroxide complexes, the better the metal will be removed.

Factors affecting phenol uptake from aqueous solution by carbon (CS-55) are investigated thoroughly. In this concern, it is found that the equilibrium was attained within 48 h. The maximum uptake has been occurred in pH neutral region (6-8). It is found that the adsorptive capacity, q^0, for phenol is $\simeq$ 53 mg / g according to the Langmuir adsorption isotherm. The substituent group attached to the phenol molecule has a measurable influence on the adsorption capacity and follows the orders: p-nitrophenol > p-chlorophenol > p-methylphenol > phenol . This is referred to the solubility and molecular structure of these solutes where the adsorbability increases as the solubility decreases .

In second section, the competitive adsorption of the three binary systems (phenol + UO_2^{2+} ; phenol + Cu^{2+} and phenol + Zn^{2+}) depends entirely on the solution pH and the equilibrium time. This observation can be explained on the basis of the fast kinetics of metal ions (equilibrium takes place within 3-4 h.), compared to phenol compound (equilibrium of phenol takes place after 48 h.). Thus, the metal ions is adsorbed first before phenol molecule. This is due to the fact that the surface of carbon (CS-55) has certain active sites preferable for metal uptake and these sites constitute the backbone of the metal uptake (Si-OH). At the same time, there are special sites on carbon attributed to the oxygen surface groups which are formed during activation procedures and these groups are available for both phenol molecule and metal ions. These sites are used by metal at first because of its fast kinetics and according to the solution pH. This means that, the uptake of phenol affected by metal uptake. It was clear that, the surface chemistry of the activated carbon is effective in metal uptake than its surface area or its porosity.

Part 3. Shows the continuous- column mode experiments, and it includes two sections.

In the first section, the breakthrough curves of phenol using carbon (CS-55) were described. It was found that, by reducing the flow rate, the service time increased and hence the volume of phenol treated effectively increased. When the bed depth is increased, the number of sorption sites is also increased. Thus, a large bed depth will take a longer time to become saturated with a similar amount of pollutant at a constant flow rate. The volume treated at breakpoint increases as the concentration of phenol decreases. The second group in p- position with phenol molecule seems to have some effect on the column adsorption capacity. This can be clearly where early breakthrough of phenol occurs at first followed by p- methylphenol, p- chlorophenol, and p- nitrophenol respectively.

In the second Section, the binary system (phenol + UO_2^{2+}) was examined using carbon (CS- 55) columns. It was clear that the (CS-55) column was successfully regenerated using $\simeq$ 1 L 0.1 N H NO_3 rinse followed by 1L of 0.1 N NaOH rinse. Column pH is the critical parameter influencing column performance. When the regeneration step was used on virgin carbon, a dramatic improvement in the column performance was observed. This was attributed to an increase in OH^- available for surface as well as an increase in the number of surface sites available for adsorption.

An application of using carbon (CS-55) for removing some impurities of crude phosphoric acid was examined. The acid (P_2O_5 = 48.42%) is kindly supplied by Abu-Zaabal Co., Egypt. The results prove that the ability of using carbon (CS-55) in high acidic medium ($\simeq$5 M H_3 PO_4) in batch mode and its efficiency for reduction the concentration of uranium, nickel, cooper and zinc as well as the organic content.