A Study of the Structural Features of Metal Impregnated Charcoals by X-Ray Diffraction Technique

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Summary: Activated charcoal was impregnated with cobalt, nickel, copper and zinc. The impregnated charcoals have been studied employing x-ray diffraction technique. Various structural parameters including crystallite size, degree of amorphousness, radius of gyration and density have been calculated from the x-ray diffraction patterns. The results reveal that structural properties of charcoal change upon impregnation with metal ions.

Introduction

Activated charcoals are extensively used as adsorbent [1-3], catalyst [4,5] and catalyst support [6]. The adsorbent properties of activated charcoals are essentially attributed to their large surface area, high degree of surface reactivity, universal adsorption effect and the favorable pore size distribution which makes the internal surface accessible thus enhancing adsorption rate and mechanical strength [7]. The properties of activated charcoal can be modified by the addition of metals which have profound effect on both the reactivity and selectivity of the surface in the catalytic reaction [8]. The metal supported, charcoal behaves as a catalyst in many chemical reactions. In this work the activated charcoal samples were modified by metal impregnation and their structural properties studied by x-ray diffraction. The uses of these materials as adsorbents, have already been reported [9].

Results and Discussion

X-ray diffraction patterns of virgin and metal (Co, Ni, Cu and Zn) impregnated charcoals are reproduced in Figure 1. It is evident from diffused halos at low 2θ values that all these samples are predominantly amorphous in nature [10]. The XRD patterns can be utilized to determine various parameters [11,12] which throw some light on the structural properties of the material. The results are presented in Table-1.

The d spacing i.e., interlayer spacing remains more or less the same. Slight decrease is observed for zinc impregnated charcoal samples due to the shifting in the (002) peak in the diffractogram. The densities of the metal impregnated samples were calculated [13]. The results reveal that upon metal impregnation the density of sample is enhanced, which may be attributed to the presence of metal atoms in the sample that cause an increase in weight of the sample.

Table-1: Determined values of different parameters of metal impregnated charcoal

Sample	ρ	DOA	Lc	Ro
Charcoal	2.16	8.45	8.33	0.960
Co - C	2.16	5.80	13.58	0.477
No - C	2.18	6.40	12.32	0.890
Cu - C	2.17	7.35	10.72	0.615
Zn - C	2.24	5.30	14.89	1.151

The degree of amorphousness (D.O.A) can be obtained for qualitative comparison by an arbitrary measure of the radial intensity distribution on the assumption that more amorphous the material broader will be the halo [10,14]. The radial width at half maximum intensity when used as a parameter for comparison shows that a decrease in D.O.A. occurs upon metal impregnation of charcoal.

The samples are not completely amorphous. This is evident from the diffuse halos having small triangular humps. This small degree of crystallinity is measured as 'mean defect distance' or mean crystallite size (Lc). Lc was calculated by the method of Short and Walker [15]. The results presented in Table-1 show that the Lc increase upon metal impregnation of charcoal.

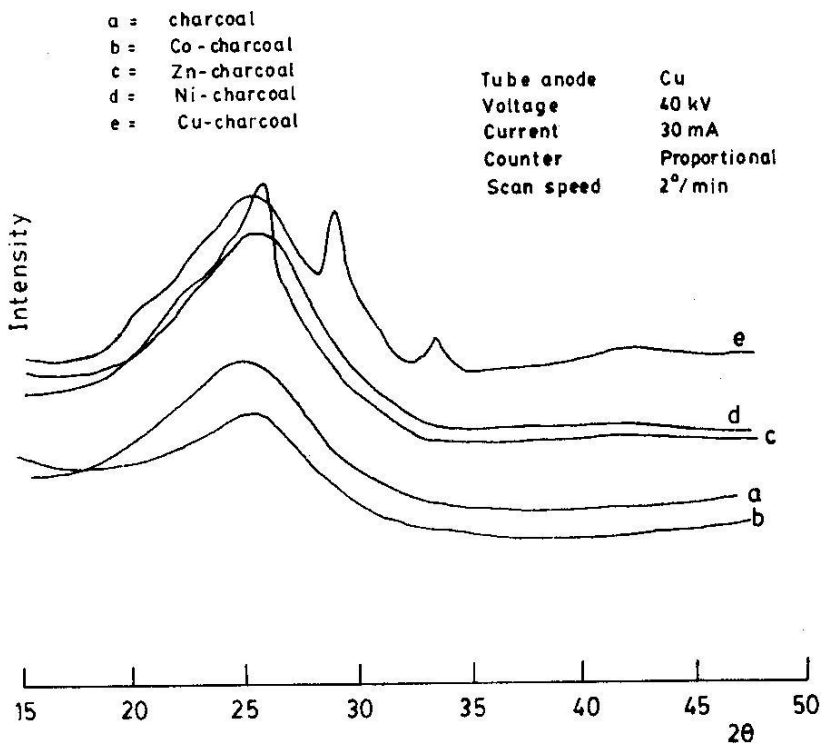

Fig. 1: XRD patterns of charcoal and metal impregnated charcoals.

The radius of gyration (Ro) was calculated from Guinier equation [12].

$$\ln I = \ln Io - h\,Ro/3$$

where; Io and I are intensities of undiffracted and diffracted x-rays respectively, $h = 4\pi\sin\theta/\lambda$ and Ro is radius of gyration in nm. The radius of gyration decreases upon metal impregnation. It is discernible from the present discussion that the structural parameters determined for copper impregnated charcoal are different from other samples. A close look at the diffraction pattern of the sample reveals that unreacted copper nitrate is present in the sample. The diffraction peaks at d values 2.14 and 3.02 are indicative of the presence of copper nitrate.

Experimental

Chemicals

The chemicals used in the present studies include activated charcoal (BDH, item No. 33032), Cobaltous nitrate hexahydrate (Fluka, item No. 60833), nickel (II) nitrate trihydrate (Fluka, item No. 61197), cupric nitrate trihydrate (Fluka item No. 61197) and zinc nitrate hexahydrate (Fluka, item No. 96482).

Preparation of impregnated charcoal

5g of activated charcoal were soaked in 100 ml of 0.2% (W/V) metal nitrate solutions. After 8h the solution was slowly evaporated at 100°C to a slurry. The mixtures were then dried at 350°C for 4h in a vacuum oven. A blank charcoal sample was prepared by adopting similar procedure. In the blank sample, distilled water was used instead of metal nitrate solutions.

X-ray diffraction

Diffraction patterns of the samples were obtained on a Philips PW 1060/70 diffractometer. The detector was an argon filled proportional counter linked to a PW 1390 rate meter and channel analyzer. The radiation was generated at 1.5418Å from a PW 1730 generator operated at 40kV and 30 mA.

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