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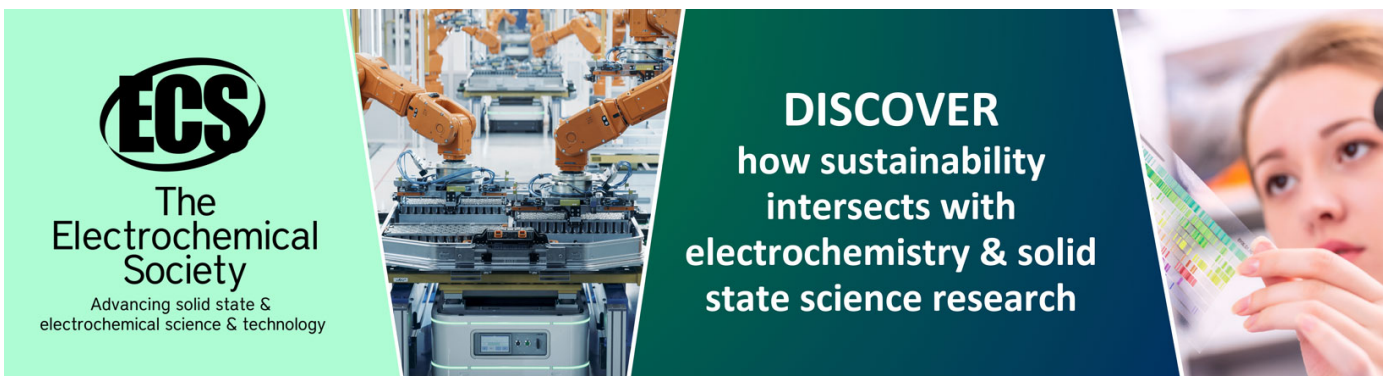
Structural characterization of graphene layers in various Indian coals by X-Ray Diffraction technique

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Structural characterization of graphene layers in various Indian coals by X-Ray Diffraction technique

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Abstract. The results of the structural investigation of three Indian coals showed that, the structural parameters like fa & Lc increased where as interlayer spacing d_{002} decreased with increase in carbon content, aromaticity and coal rank. These structural parameters change just opposite with increase in volatile matter content. Considering the ‘turbostratic’ structure for coals, the minimum separation between aromatic lamellae was found to vary between 3.34 to 3.61 Å for these coals. As the aromaticity increased, the interlayer spacing decreased an indication of more graphitization of the sample. Volatile matter and carbon content had a strong influence on the aromaticity, interlayer spacing and stacking height on the sample. The average number of carbon atoms per aromatic lamellae and number of layers in the lamellae was found to be 16-21 and 7-8 for all the samples.

KEYWORDS: Coal, Graphene layer, Aromaticity, Turbostratic structure.

1. Introduction

Allotropes of carbon have been a subject of great interest for researchers several years. Of these best known are graphite, diamond and amorphous carbon. Amorphous carbon is the name used for coal, soot and other impure forms of the element. Amorphous carbon has a wide range of properties that are primarily controlled by the different bond hybridizations possible in such materials. Graphite consists purely of sp^2 hybridized bonds, whereas diamond like carbon is a metastable form of amorphous carbon containing significant fraction of sp^3 bonds. This gives different properties like high strength, flexibility etc. Due to these properties they are used in thin film technology and in nanoscale electronic devices. The existence of crystallites in coal structure is confirmed by the appearance of the peaks corresponding to the (002), (100) and (110) reflections of graphite in coal. It is suggested that carbon in coal has an intermediate structure called turbostratic between graphite and amorphous structure. Coal also contains significant amount of highly disordered material, amorphous carbon, which is responsible for the background intensity of the diffractions [1-9].



The aim of the present study is to determine the stacking structure of selected coal samples with carbon content of 72 – 77.4%. The parameters include the interlayer spacing and crystalline size along the c-axis (L_c) and in the layer sheet direction (L_a), the average number of carbon atom (n) per aromatic lamellae and packing.

2. Experimental

The samples were de-mineralized with strong base like NaOH and KOH to avoid the effect of mineral matter on the quantitative analysis before X-ray diffraction studies [1-4]. From the finely powdered coal about 10g of the sample was dispersed in 50ml of NaOH and KOH separately and the mixture was stirred for 1hr at 27°C. Finally the treated coal was washed and dried in the air at 70°C for 3 hrs and allowed to cool slowly in a dessicator.

2.1. X-ray diffraction Analysis

The XRD data collection was performed by a Bruker AXS D8 Advance X-ray powder Diffractometer. The broad hump in this region was fitted with two Gaussian peaks around 20° and 26°, namely γ - band and Π -band (d_{002}) respectively. Theoretically, the areas under the γ and Π -peaks are believed to be equal to the number of aromatic carbon atoms (C_{ar}) and aliphatic carbon atoms (C_{al}) respectively [1-6].

3. Result and Discussion

The X-ray diffraction profile of the demineralized coal sample is shown in Figure 1. The samples exhibited high background intensity indicating that the coals contained a proportion of highly disordered materials in the form of amorphous carbon [1-13].

The diffraction profiles show the presence of a clear asymmetric (002) band around $\sim 25.5^\circ$, which suggests the existence of another band (γ) on its left. The (γ) band around 20° was reported by many authors [1-9, 11-13]. It was attributed to the presence of saturated structures such aliphatic side chains, attached to the edge of the coal crystallites. The (002) band indicates the spacing of aromatic ring layer, while (γ) band reflects the packing distance of saturated structures. In addition, the coals also contained some graphite-like structures (crystalline carbon) indicated by the presence of a clear (002) band at $\sim 25.5^\circ$ and (10) weak band at $\sim 42.3^\circ$. These observations suggest that, the crystallites in all the coal samples have intermediate structures between graphite and amorphous state called turbostratic structure or random layer lattice structure..

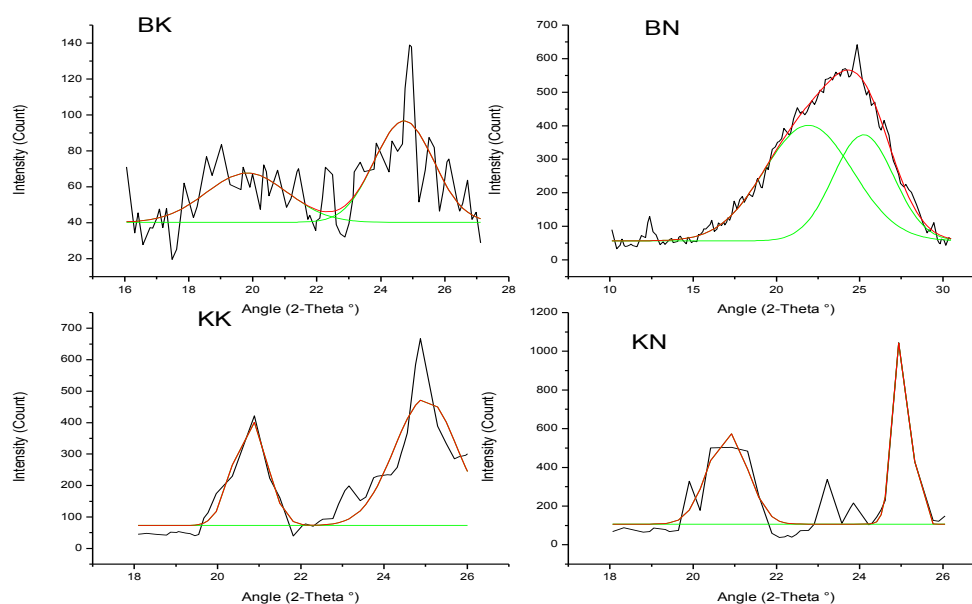


Fig.1. XRD pattern of demineralized coal sample with NaOH and KOH (BN and KN)

3.2. Determination of number of carbon atom (n) and packing aromatic lamellae

The average number of carbon atoms (n) per aromatic lamellae was calculated from the equation given elsewhere [3-4, 11-14]. It was found to be varying from 16 to 21 and also increasing with increase of coal rank. This result is in good agreement with the study carried out by Binoy et al. [2] in Assam coals. There was a high degree of correlation exist between these two parameters ($R^2=0.99$).

Tuinstra and Koenig [10] noted that the intensity ratio of the D and G modes, I_D/I_G , varies inversely with the in-plane correlation length or grain size of the graphite. This equation was based on the Raman spectrum graphite which is highly ordered carbon. In the present study, the intensity ratio I_{20}/I_{26} from X-ray diffraction, is plotted against La values and is shown in Fig.2. It is found that, the sample is agreeing with Tuistra-Koenig relationship for nano crystalline Carbon. The I_{20}/I_{26} ratio is proportional to the number of rings at the edge of the grain. As the La increases, the intensity ratio decreases quickly.

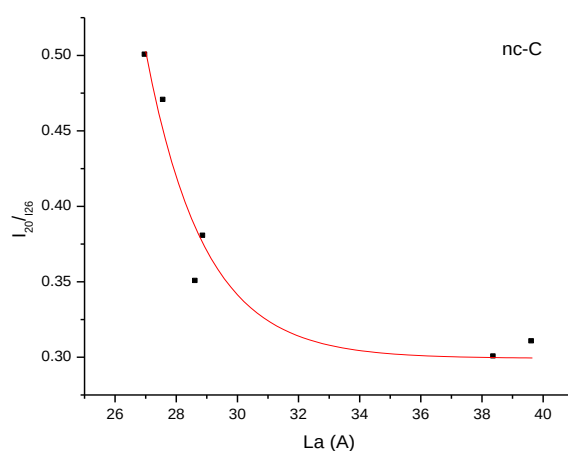


Fig.2. Relation between L_c and intensity ratio I_{20}/I_{26}

3.3. Packing of aromatic –lamellae

The average interlayer distance of the lamellae were computed from the position of ‘002’ reflection, where as the average dimension of the packets of the lamellae in the direction perpendicular to their planes were estimated from its half width [12-13]. The number of layer as determined by this method using L_c was found to be 7 to 8 for coal samples. The aromaticity (fa) and rank (I_{26}/I_{20}) ratios for the samples ranged from 0.61 to 0.71 and 2.007 to 3.275, respectively. The average lateral sizes (La) and average stacking heights (L_c) of the layer structures in the coals samples, measured using the Scherrer equation ranged from 27.61 Å to 38.43 Å and 23.73 Å to 20.16 Å respectively. Interlayer spacing (d_{002}) of the crystallite structure ranges from 3.60 Å to 3.34 Å. The percentage of ordered carbon and the degree of parallel stacking of the lamellae increased with rank of the sample. The volatile matter (V.M) changed from 36.2 wt % to 24.9 wt% where as oxygen content varied from 21.37-16.64 wt% for the studied samples

Conclusions

The results of the structural investigation of three Indian coals showed that the structural parameters like fa & L_c increased with increase in carbon content where as interlayer spacing d_{002} decreased with increase in carbon content, aromaticity and coal rank. These structural parameters change just opposite with increase in volatile matter content. Considering the ‘turbostratic’ structure for coals, the minimum separation between aromatic lamellae was found to vary between 3.34 to 3.61 Å for these coals. As the aromaticity increased, the interlayer spacing decreased an indication of more graphitization of the sample. Volatile matter and carbon content had a strong influence on the aromaticity, interlayer spacing and

stacking height on the sample. The average number of carbon atoms per aromatic lamellae and number of layers in the lamellae was found to be 16-21 and 7-8 for all the samples. The carbon in coal is having the structure of nanocrystalline carbon.

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