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Characterization of Annealed Activated Carbon

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Activated carbon is peppered with low volume pores that increase its surface area, thereby increasing the area for adsorption or chemical reactions. After annealing activated carbon at 2300 ^{0}C, the appearance is noticeably altered by the development of cigar-shaped microrods appearing on microcystallites, Figure 1a[1] High resolution transmission electron microscopy (HRTEM) show the rods are more akin to graphite crystals by analysis of the selected area diffraction (SAED) patterns Figure 1b. As the process for the microrod nucleation might be inherent in the "activation" mechanism of the carbon, it is essential to characterize before and after annealing to further understand the nucleation and growth processes of these "graphite microrods."

Activated carbon, Sigma-Aldrich #161551 was annealed at 2300 ^{0}C in a helium atmosphere for 36 hours. The changes in the porosity and surface area of the annealed product were characterized on the basis of small angle X-ray scattering (SAXS) data by analysis of the Porod region. The texture of the heat treated samples was investigated by scanning electron microscopy (SEM) and HRTEM. X-ray diffraction, (XRD), Raman spectroscopy, and SAXS were used to estimate the changes in structural properties of heat-treated activated carbon samples. XPS confirmed the presence of surface elements as a result of chemisorption on the surfaces of the samples.

The activated carbon is composed of small particles, ~18.5 nm (confirmed by SAXS and SEM), agglomerated into larger particles, 2-35 μ. After annealing at 2300 ^{0}C the primary particle is still ~18.5 nm with 2-35 μ agglomerations, although the occurrence of the larger agglomerations is less by 60%, Figure 2. During annealing activated carbon leads to the formation of a "turbostratic structure."[2] As the formation of the turbostratic structure progresses, micro-pores coalesce and defects are incorporated into the graphitic structure, such as pentagons. Pentagons have been theorized as the pinned axis of a single sheet of graphite, which helically wraps round the defect axis resulting in the growth of a rod [3]. HRTEM shows that the basal plane of the rods is not parallel to the axis as in carbon fibers, it is at an angle α to the axis Figure 3a. This angle is related to the overlap angle, θ, values whereby the whorl around the axis preserves the ABAB stacking of graphite [3].

$$\sin\frac{2\alpha}{2} = \frac{2\pi - \theta}{2\pi}$$

There is evidence of pentagon inclusion in the activated carbon, before and after annealing as seen in HRTEM images of defects, Figure 3b [4].

References:

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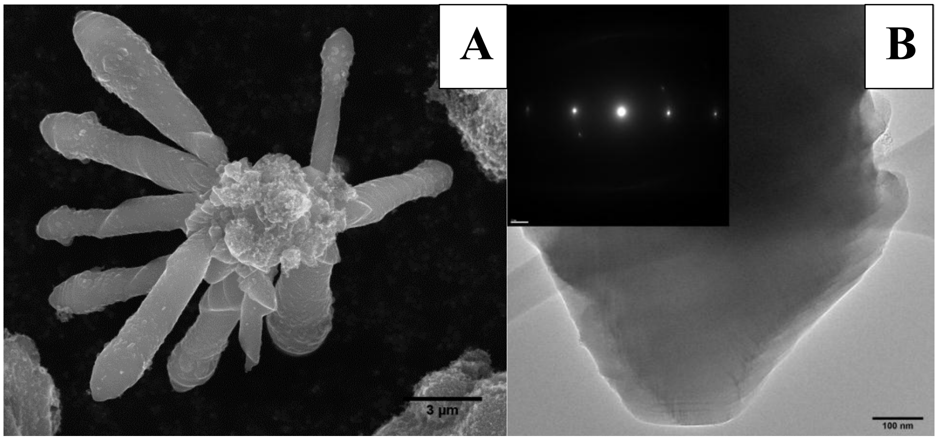

Figure 1. A) Micorod on Activated Carbon treated 2300 ^{o}C, B) Rod tip and SAED of rod tip, (002) and (004) diffraction

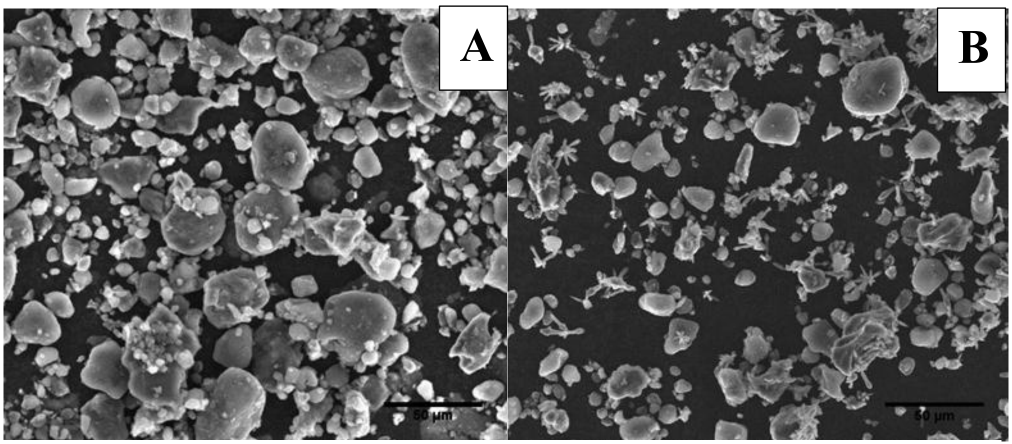

Figure 2. A) Activated Carbon as received, B) Activated carbon annealed at 2300 ^{o}C in a He atmosphere for 36 h

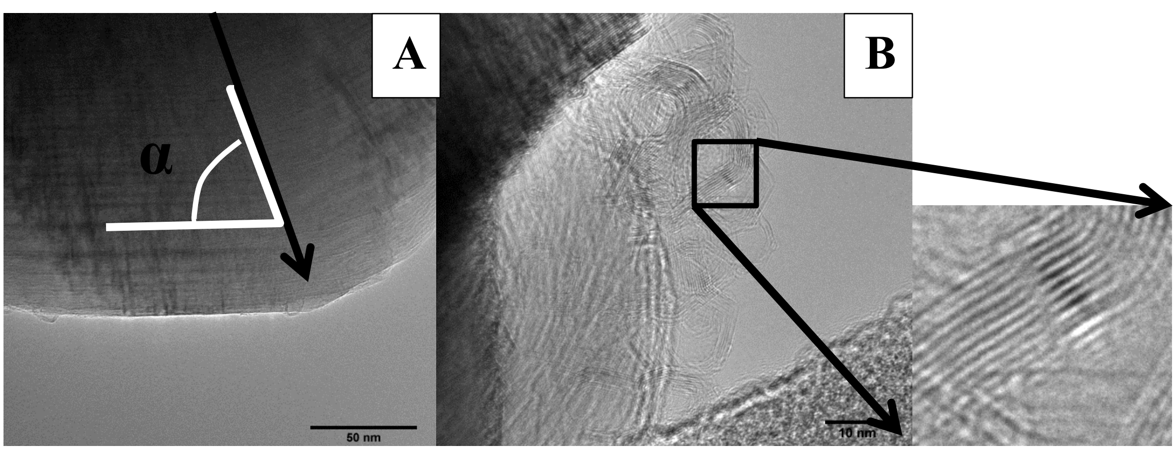

Figure 3. A) TEM of a microrod tip with axis shown by the black arrow and basal planes at $\alpha = 75^{o}$, B) Defects in the activated carbon particle annealed at 2300 ^{o}C

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