Poster E11

Generation of Carbon Microparticles by Hydrothermal Conversion

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Our study investigates a new valorization way of black liquor (BL), an alcaline aqueous residue derived from wood cooking. This solution contains mainly dissolved lignin, hydrolyzed part of cellulose/hemicellulose and different salts of Na, Ca, K, sulfide, sulfate,... BL dry matter represents 23wt% with a high organic content ($\sim$ 100g.L^{-1} of carbon). Currently, BL is burnt to produce heat with salts recuperation in paper industry. In general, this technological step controls (limit) paper production. The objective of our research is to provide alternative more valuable valorization of the organic matter content of BL by using its hydrothermal conversion under sub /supercritical conditions. Indeed, water, around its critical point (T=374°C, P=22,1MPa) has special physical and consequently reactive properties [1]. As a result, BL is converted. A preliminary work has shown an interesting way of valorization by generation of carbon microparticles. Our paper characterizes the formed solid phase in regard to the operating conditions of the batch hydrothermal conversion of BL solutions.

The experiments were performed in batch process (5mL), to prevent salts precipitation, at 350°C and 25MPa. Studied parameters were: BL concentration (10wt% -100wt%), reaction time (15min-15h), heating and cooling rates. All experiments generated three phases. After separation, they were rapidly analyzed. The solids were analysed by environmental scanning electron microscopy, X-ray diffraction, infrared spectroscopy, Raman spectroscopy, Dynamic Vapor Sorption.

Although the amount of solid was low ($\sim$ 0.3wt% of dry BL solution mass), the proportion of carbon recovered in the solid phase is $\sim$ 13wt%. The solid residue is mainly composed by carbon and oxygen and the FTIR analyse suggests it comes from the phenolics precipitation. Its structure is complex, varying from aggregates of micrometric spherical particles (0.4-1 μm or 1-11 μm) to denser more massif structure showing an external porosity. Under certain operating conditions, an internal porosity was also highlighted for long reaction time. DVS analyse suggests a modification of the surface. One of the valorization opportunities for the microspheres obtained by hydrothermal conversion could be the fabrication of hollow sphere containing inorganic compounds [2].

References

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[2] SEVILLA, M., FUERTES, A., Chemistry A European Journal, Vol.15, **2009**, p.4195