Surface modification of pyrolytic carbon

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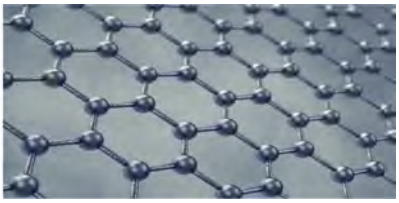
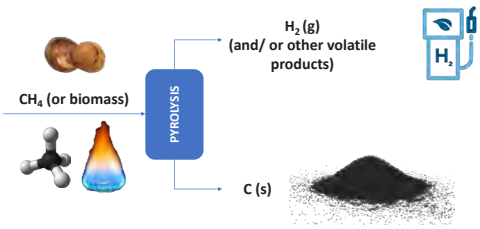
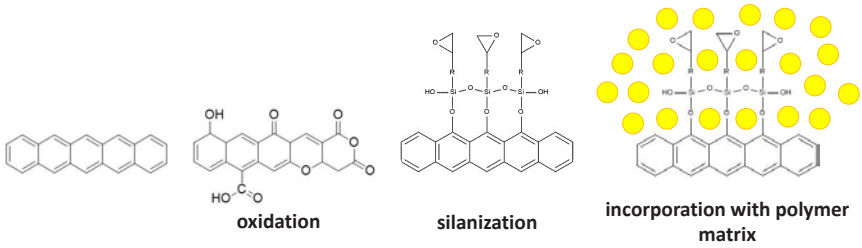


FUNDAMENTAL GOAL

Surface modification of pyrolytic carbon obtained from natural gas and biomass to make polymer composites with resins, so it could be used as e.g., a building material with dual function: lightweight and durable material that can be used in the first step of wastewater and waste air treatment (building material for dumps and buildings).[1]

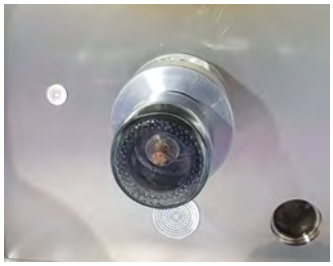




EXPERIMENT

Carbon black obtained from pyrolysis of waste biomass-coconut shells (CSC), was underwent experiments of (I) **oxidation** (both wet and dry (plasma treatment)), (II) **hydration and reduction**, (III) **silanization** and (IV) **functionalization with hydrazines**. In the experiments of wet oxidation (solvothermal oxidation in autoclaves) the mixture of pyrolytic carbon and HNO_3 (aq) were mixed for 2h and at 120 °C, filtered and dried overnight. **Cold plasma-treated carbons** were exposed to the mixture of the ionised **Ar** and $\mathbf{O_2}$ under high vacuum and power dosages of 300 W and 600 W. In experiments of hydration of oxidized carbon, its surface was subsequentially treated in very diluted H_2SO_4 (aq) and with $LiAlH_4$ to achieve more desired hydroxyl groups (-OH). The hydrated and reduced oxidized carbon samples were furtherly functionalized with (3-aminopropyl)trimetoxysilane (**APTMS**) and 4-fluoro-phenylhydrazine (**FNP**) for 6h and 24h at 80 °C. These samples were washed by centrifugation with the proper solvent and dried overnight.

Instrumental set-up for oxidation of carbon in autoclaves with jacket (left) and without jacket (right)



Instrumental set-up for plasma oxidation of carbon

RESULTS

The **XPS** results that functionalization with both, silanes and hydrazine was successful. In the case of silanization with APTMS, the found amount of **Si** on the CSC surface was **4,95 %** and in the case of the functionalization with FNP amount of **F** was **2,23 %**. From the observed **ATR** spectra of functionalized carbons can be seen that in silanized CSC, the desired **Si-O-C** bond was found on **1100 cm^{-1}**. On the other side, ATR spectra of hydrazine functionalized CSC indicated that **C-F** bond was created and detected on the position of **810 cm^{-1}**. For performed functionalizations, the surface of the carbon was preserved.

CONCLUSION

Overall surface modification of the CSC with APTMS and FNP was shown to be successful. The use of autoclaves, i.e. solvothermal reactions was shown the best for carrying reactions where there is a need for good control of the temperature and pressure. Further experiments will be focused on surface modification with simple amines (e.g., ethylenediamine, EDA) and mixing of the chosen polymer systems (e.g., epoxy and polyurethane resins) with functionalized carbons to make carbon composites.

Refrenece: 1. Pustahija, L.; Kern, W. Surface Functionalization of (Pyrolytic) Carbon—An Overview. C 2023, 9, 38. https://doi.org/10.3390/c9020038

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