

https://dspace.org/

Development of mixed matrix membrane for desulfurization of sulfur rich petroleum stream

Burla, Swathi

2022

https://hdl.handle.net/10539/34379

Downloaded from a DSpace repository. https://dspace.org/

ABSTRACT

The presented study focused on the methods of synthesizing the hexagonal boron nitride (h-BN), carbon dots (C-dots) and carbon dot doped boron nitride (CBN) nano materials for applications in dibenzothiophene (DBT) desulfurization. The synthesized materials were characterized using different analytical techniques such as transmission electron microscopy (TEM), scanning electron microscope (SEM), X-ray diffraction (XRD) and fourier transform infra-red (FTIR) to confirm the synthesis of the desired products. H-BN was synthesized using chemical vapor deposition (CVD) and heat treatment furnace (HTF) methods, with CVD producing better h-BN product at 900 ^{o}C calcination temperature and HTF products forming with high impurities. FTIR analysis of h-BN CVD (900 ^{o}C) showed two characteristic peaks at 1369 cm^{-1} and 787 cm^{-1} confirming the formation of h-BN. An extra peak at 3250 cm^{-1} of hydroxyl (-OH) vibration, indicates the presence of small amount of -OH on h-BN on all the products. The -OH peak intensity was decreased with increased calcination temperature, as shown from the FTIR spectra of different h-BN products calcined at 3 different temperatures. The TEM analysis observed several stacked parallel line-like structures (layers) with multiple layered disordered structures of h-BN flakes. The SEM image displayed an unevenly distributed grained powder of h-BN microstructure. XRD peaks of h-BN synthesized via CVD method reveals a strong peak of the crystallographic (002) plane at $2\theta = 27.17°$ with some broadening, indicating low crystalline nature of h-BN. The C-dots were successfully synthesized using hydrothermal carbonization of chitosan raw material in an autoclave. The existence of various functional groups in C-dots was confirmed by the FTIR spectroscopy. TEM images clearly showed a nearly spherical shaped C-dots with diameter of 5-10 nm. XRD analysis showed a broad diffraction peak at $2\theta = 21.07°$ indicating the presence of amorphous carbon.

The synthesis of novel CBN material was successfully carried out by adapting a method from the literature. The FTIR analysis shows that the h-BN was not modified by C-dot addition. TEM image of CBN confirms the doping of nano sized C-dots into the h-BN lateral layers. The SEM images reveal porous microstructure of the CBN nanomaterial indicating the doping of C-dots induced some structural defects into the h-BN that resulted in reduction of the crystalline nature of CBN. XRD analysis of CBN revealed a slight shift in main peak as a result of amorphous C-dot doping on h-BN.

Mixed matrix membrane (MMM) membranes based on polyphenylsulfone (PPSU) embedded with the synthesized h-BN and CBN were developed and analysed for the desulfurization of DBT-hexane model fuel. The FTIR analysis of the membranes revealed that there was no change in the PPSU functionalities except for the additional peak at 3233 cm^{-1} for PPSU-CBN membrane. This indicates the presence of -OH group which might result in the interaction between CBN and PPSU of the MMM membranes. The SEM images of both pristine PPSU and PPSU MMM membranes revealed the porous nature of the developed membranes. The PPSU membrane showed less pores compared to the MMMs. The adsorption studies at varying parameters show that PPSU-CBN membranes were performing better than the PPSU-h-BN membranes. The addition of C-dots is believed to have increased the adsorption sites in the CBN during synthesis which enhanced the adsorption of DBT on membranes. The temperature variation had more influence on the adsorption capacity of the membranes which can be observed from the adsorption isotherm studies. The kinetic models of the adsorption are studied for the PPSU-CBN membrane adsorption system. The models show that the diffusion of adsorbate into the pore of the adsorbent and the interaction of adsorbate-adsorbent are favoured at room temperature. The results indicate a multi mechanism occurring at 309.15 K where the reaction rate was considered as a controlling factor. Based on the R^2 values, it can be concluded that the removal of sulfur using a polymeric membrane embedded with CBN involved various

mechanisms and the diffusion film models confirmed that the adsorption was controlled by the membrane-pore diffusion mechanism.