Dispersive liquid-liquid microextraction using magnetic nanoparticles for preconcentration of some pollutants aqueous samples

Adineh Aminianfar*,

In recent years, synthetic dyes are extensively used in various industries. Rhodamine B (RB) belongs to a class of synthetic xanthine dyes that is toxic and carcinogenic for humans and animals and prohibited in foodstuffs all over the world. In the first part of the study, sulfonic acid functional groups were created onto the surface of synthesized KIT-6 mesoporous magnetite nanoparticles to form Fe3O4@SiO2@KIT-6-SO3H as a recoverable adsorbent for efficient removal of RB cationic dye aqueous solutions. For this purpose, magnetite nanoparticles (MNPs) were synthesized via chemical precipitation method. The surface of bare MNPs was coated with SiO2 layar and then by a highly ordered large pore silica mesoporous KIT-6. After that, mesoporous MNPs were functionalized with SO3H groups. The synthesized nanocomposits were characterized by SEM, FT-IR, XRD and EDX methods. The various parameters affecting dye removal such as the amount of adsorbent, pH of solution, salt effect, volume of sample and contact time were evaluated using L16 Taguchi experimental design method. Under optimal conditions (sample volume: 10 mL, pH :4, sorbent mass: 0.08 g, NaCl content: 0.005 mol L-1and contact time: 15 min) the RB removal was reached as 95%. Adsorption data were studied using Langmuir, Freundlich and Temkin models. Also, the kinetic studies showed the dye adsorption followed well with the pseudo-second order kinetic model. Ultimately, the efficiency of the synthesized adsorbent for dye removal real water samples was successfully confirmed. The results of this study can be used to design an appropriate adsorption treatment plan for removal of Rhodamine B wastewater samples. In the second part, an efficient preconcentration technique was developed for determination of trace amounts of RB in aqueous samples. In the proposed coupled magnetic solid phase extraction - dispersive liquid - liquid microextraction method

(MSPE-DLLME), a new nanoparticle, Fe3O4@SiO2@KIT-6-SO3H, was synthesized, characterized and introduced as a sorbent for preconcentration of RB as a cationic dye in aqueous samples. The adsorbed RB was desorbed by acidic ethanol the surface of magnetic nanoparticles during the MSPE process and then, the desorbed solution and the extraction solvent were injected into the distilled water in a conical tube for next DLLME process. After centrifuge of the cloudy solution, sedimented solvent was diluted with ethanol and determined by UV-Vis spectrophotometry. Some experimental parameters such as the amount of adsorbent, salt concentration, pH of solution, type and volume of acidic dispersive solvent, type and volume of extraction solvent, strength of acid and volume of distilled water were investigated and optimized in order to have higher extraction efficiency. Under the optimized conditions, the relative standard deviation and the limit of detection (LOD) of the proposed method were obtained as 6.8% and 0.2 µg L-1, respectively. The enhancement factor was 35 and the linearity was obtained in the range of 1-300 µgL-1 with the correlation coefficient of 0.9931. Based on the results, it seems the proposed MSPE-DLLME method is a good way to preconcentrate and determine trace amounts of RB in aqueous samples.

Keywords : Magnetic nanoparticles, Rhodamine B, Adsorption, Silica mesoporous KIT-6, Solid phase extraction, Dispersive liquid – liquid microextraction