



# Enhancement of oil recovery using zirconium-chitosan hybrid composite by adsorptive method



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## ABSTRACT

Recovery of oil from oil-in-water emulsion has been investigated by many scientists and it continues to be a challenging task for environmental scientists so far. Among all the techniques, adsorption is found to be an appropriate process for the removal of oil from oil-in-water emulsion owing to its high efficiency and easy operation. A hybrid material, zirconium-chitosan composite (Zr-CS-HC) was prepared to remove the oil from oil-in-water emulsion and oil was measured by extractive gravimetric method. Various parameters viz., agitation time, pH, sorbent dosage and initial oil concentration for maximum sorption were optimized. In this study, the maximum oil removal percentage was found to be at pH 3.0 and a minimum contact time of 50 min using prepared sorbent. The pH of the sorption studies revealed that oil sorption was favored in acidic condition. The sorbent was characterized using FTIR, SEM with EDAX, XRD, TGA and DSC; contact angle and heat of combustion. The experimental data were explained using Freundlich, Langmuir, D–R and Tempkin isotherms to find the best fit for the sorption process. Thermodynamic parameters such as  $\Delta G^\circ$ ,  $\Delta H^\circ$  and  $\Delta S^\circ$  were calculated to understand the nature of sorption process. This work provides a potential platform for the expansion of oil removal technology.

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## 1. Introduction

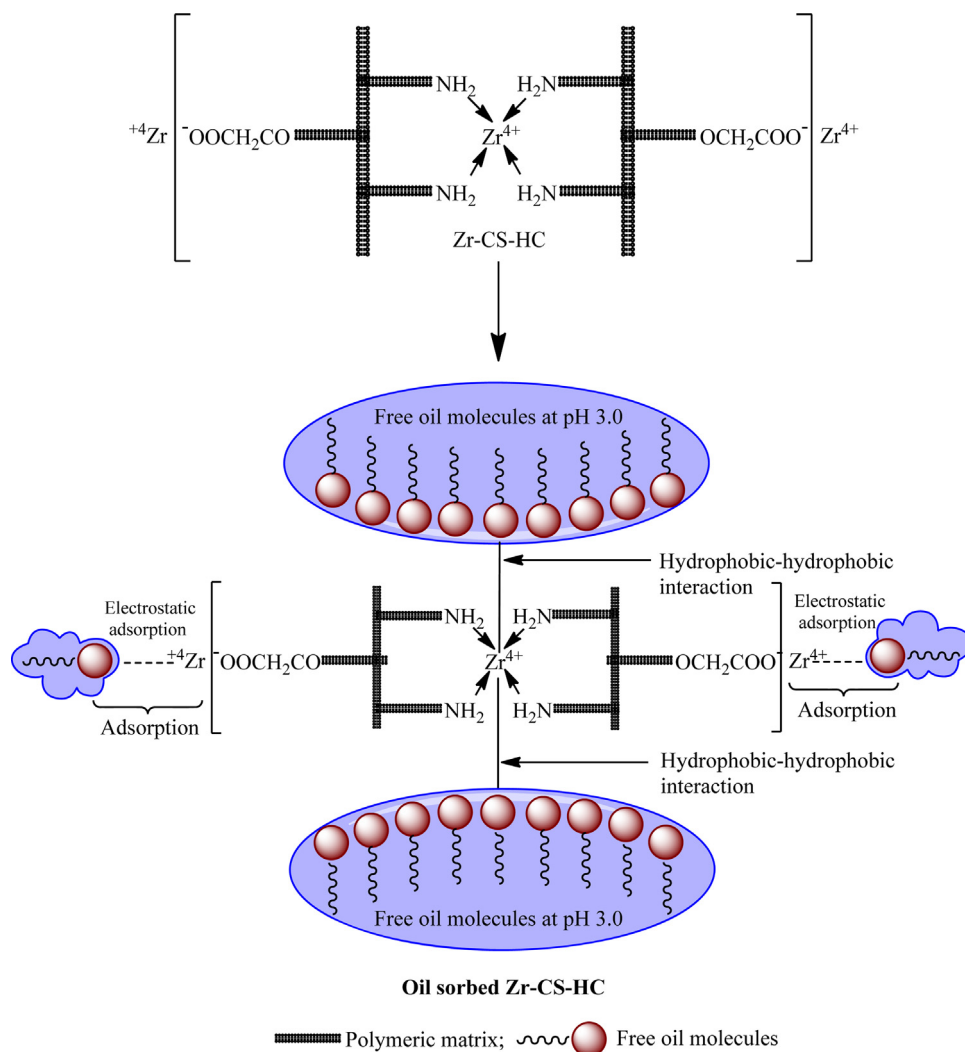
Oil is an essential resource in energy, food and chemical industries, and it is being widely utilized in our lifestyle. However, many of the industries cause the hazard of oil pollution by directly discharging their effluents into the surrounding environment. The oil-in-water emulsion is found in most of the industrial effluents discharged during the machining process such as drilling, grinding, cutting, milling, etc. Many industrial effluents contain mixture of hydrocarbons in the form of oily wastewater and are poorly biodegradable (Scholz & Fuchs, 2000). Removal of oil in the form of stable oil-in-water emulsion is one of the imperative environmental concerns as a result of increasing growth of engineering products throughout the world (Sivasurian, Elanchezhiyan, & Meenakshi, 2015). Oil present in wastewater can be found in several types like fats, lubricants, cutting fluids, light hydrocarbons and heavy hydrocarbons (Srinivasan & Viraghavan, 2008). Though World Health Organization (WHO) has not mentioned the optimum limit for oil and grease, the Ministry of Environment and Forests, Government of India has prescribed the discharge of wastewater

quality with oil concentration not exceeding 5 mg/L (MoEF, 2008). Majority of the effluents contain oil-in-water emulsion as basic contaminants. Even very low oil concentrations will cause serious ecological problems to the environment owing to the toxic and hazardous properties of its components (Srinivasan & Viraghavan, 2010). Hence, it is necessary to find out green technology which is effective and economical in removing oil from industrial wastewater before discharging them into the environment.

A number of remarkable techniques have been suggested for the removal of oil from oil-in-water emulsion in the literature so far, such as adsorption (Ahmad, Sumathi, & Hameed, 2005; Li, Zhu, Li, Na, & Wang, 2013; Ibrahim, Ang, & Wang, 2009; Li et al., 2013; Long et al., 2012; Rajakovi & Rajakovi, 2008), flocculation (Pinotti, Bevilacqua, & Zaritzky, 2001), coagulation (Ahmad, Sumathi, & Hameed, 2006), flotation (Moosai & Dawe, 2003), membrane technique (Scott, Jachuck, & Hall, 2001), electro-coagulation (Tir & Mostefa, 2008), electro-flotation (Vasudevan & Oturan, 2014), etc., owing to its toxicity, biological method is difficult to use for the treatment of oily wastewater. Among various chemical and physical methods, adsorption has shown to be the most beneficial and most commonly used method as it is a simple process, highly efficient, easy recovery, reusability of the adsorbent, eco-friendly and low cost because of this reason, adsorption has been the method of choice for most of the researchers, and various materials have been

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**Scheme 1.** Mechanism of oil sorption using Zr-CS-HC.

tried for the treatment of oily wastewater such as chitosan (Ahmad et al., 2005, 2006; Ibrahim et al., 2009; Piyamongkala, Mekasut, & Pongstabodee, 2008), activated carbon (Inagaki, Kawahara, Nishi, & Iwashita, 2002), coal (Xiaobing, Chunjuan, & Jiongtian, 2010), walnut shell (Srinivasan & Viraghavan, 2008), sawdust (Cambiella et al., 2006), vermiculite (Singh, Kendall, Hake, & Ramkumar, 2013), barley straw (Ibrahim et al., 2009) corn straw (Li, Zhu, Li, Na, & Wang, 2013; Li et al., 2013) raw cotton (Singh et al., 2013) organoclay (Salehi, Mowla, & Karimi, 2012) and mixture of Ca and Mg (Solisio, Lodi, Converti, & Borghi, 2002). Biopolymers have been proved as suitable sorbents for the removal of pollutants from various water systems by many researchers. Among these, chitosan has received a lot of attention and it is one of the most effective and acceptable biosorbent commonly used in wastewater treatment due to its multiple functional groups, biocompatibility, biodegradability, adsorption property and non-toxicity. The high content of amino groups is present in chitosan polymeric matrix and it also facilitates various chemical modifications in the polymer. Among transition metals, zirconium is an interesting element having higher affinity. Zirconium is accounted to be a biologically inert material, non-toxic as an element or in compound, and the oral toxicity is reported to be quite low. In addition, zirconium-chitosan hybrid composite could be an attractive sorbent of choice in the water treatment, as zirconium is not only non-toxic, but also good resistant to oxidant agents, acids and bases, high thermal stability and very low solu-

bility in water. Zirconium possesses better chelating ability with chitosan than the other metal ions. Due to good biocompatibility with chitosan, the possibility of zirconium leaching into the environment is significantly prevented and thereby decreasing the second contamination to environment (Liu, Hu, Wang, Zhang, & Huang, 2015). Moreover, zirconium incorporated biopolymers can increase the adsorption capacity and these have been established as highly effective sorbent in the removal of pollutants from aquatic environments because of the existence of higher positive charge in metal, chemical stability, strong affinity, binding tendency and less leaching effect on water (Viswanathan & Meenakshi, 2008; Zhang, Xia, Teng, Liu, & Zhang, 2013).

The main goal of the present work is to identify the suitable sorbent for the recovery of oil from oil-in-water emulsion with the influence of various physico-chemical parameters like contact time, pH, mass of the sorbent and initial concentration of oil-in-water emulsion. To the best of our literature survey, the oil sorption studies using zirconium-chitosan hybrid composite were not reported earlier. The Zr-CS-HC was prepared and used for the removal of oil from oil-in-water emulsion. The sorbent was characterized by FTIR, SEM with EDAX, XRD, TGA, DSC and contact angle measurement. The experimental data were fitted with adsorption isotherm models.

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