

UNIVERSITI TEKNOLOGI MARA

**SYNTHESIS OF FE-MKSF
CATALYST FOR OXIDATIVE
DEGRADATION OF METHYL
ORANGE IN HETEROGENEOUS
FENTON-LIKE REACTION**

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ABSTRACT

One of the promising and effective treatment methods in degrading organic pollutants is known to be heterogeneous Fenton-like reaction. Different types of iron-immobilized clay have attracted researchers' attention for heterogeneous Fenton-like catalyst due to its efficient use of environmentally benign chemicals, chemical process and low cost. However, detailed mechanistic insight during the catalysis which involved the formation and identification of radical species are remained ambiguous due to complex interaction of solid catalyst and organic pollutant. To address this gap, this research aims to synthesis and characterize iron-immobilized montmorillonite KSF clay (Fe-MKSF) catalyst, to optimize the operational conditions of heterogeneous Fenton-like reaction using the resultant catalysts and to investigate the detailed involvement of reactive radical species during the oxidative degradation of targeted pollutant. Series of Fe-MKSF catalysts were synthesized at varies iron molarity to clay ratio through impregnation-hydrothermal method. The catalytic activity of resultant catalyst was evaluated in heterogeneous Fenton-like reaction using methyl orange (MO) dye solution as the model pollutant. The physicochemical properties of these catalysts were characterized using nitrogen sorption, X-ray diffraction, scanning electron microscopy and thermal gravimetric analysis. The optimization of operational heterogeneous Fenton-like reaction condition was performed using the response surface methodology (RSM). Then, the presence of reactive radical species and its involvement during heterogeneous catalysis were identified through the utilization of radical scavenging agents at optimum operational condition. The Fe-MKSF catalyst at 20 iron molarity to clay ratio (Fe-MKSF-20) has displayed the highest catalytic MO removal (85%) compared to others composition. This finding can be well supported by significant increment in surface area by an order of magnitude from 52.2 m²/g (iron oxide) to 105.8 m²/g (Fe-MKSF-20). The optimum operational reaction conditions predicted by RSM using central composite design (CCD) is fitted to the second order regression model which were found at pH 2.8, 0.8 g/L of Fe-MKSF-20, 15 mM of hydrogen peroxide and initial MO concentration of 25 mg/L with the attainment of 98% MO removal. Interestingly, the prevailing radical species presence during the heterogeneous Fenton-like reaction were found to be hydroperoxyl (\bullet OOH) radicals and subsequently hydroxyl (\bullet OH) radicals. Based on the percentage of MO removal, it was suggested that approximately 91% of the \bullet OOH radicals existed at the interface of catalyst while 42% presence in bulk solution. Meanwhile, the interface \bullet OH radicals promoted only 41% of MO removal, whilst 7% by the bulk \bullet OH radicals. Hence, these findings have provide in- depth understanding on the role of MKSF in modulating catalytic activity of resultant Fe-MKSF catalyst and the detailed involvements as well as interactions between these radical species which presence during oxidative degradation of MO solution in heterogeneous Fenton-like reaction.

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