SYNTHESIS AND CHARACTERIZATION OF A CORN OIL BASED DEMULSIFIER FOR CRUDE OIL EMULSION

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We hereby declare that we have checked this thesis, and, in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Doctor of Philosophy.

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ABSTRAK

Kehadiran air dalam emulsi minyak berlaku pada pelbagai peringkat dalam proses pengeluaran dan rawatan minyak mentah. Kehadiran air menyebabkan beberapa masalah operasi seperti hakisan pada peralatan dan saluran paip. Hal ini juga merupakan salah satu ancaman alam sekitar/kesihatan yang boleh mengganggu operasi penapisan, dan secara amnya meningkatkan kos pengeluaran minyak. Oleh itu atas faktor ekonomi, alam sekitar dan pengoperasian, pengasingan air daripada minyak sebelum mengangkut atau menapis air ke proses terakhir adalah penting. Kaedah yang paling berkesan untuk mengatasi masalah ini ialah dengan menyahmulsifikasikan minyak mentah menggunakan penyahmulsifikasi kimia melalui kaedah sintesis demulsifier. Terdapat jurang kosong di dalam proses penyahmulsifikasi kimia, walaupun kaedah ini berkesan namun kebanyakan proses ini berasaskan petroleum dan polimer organik refraktori. Proses ini mendedahkan ekosistem sekeliling kepada risiko yang lebih berbahaya dan kaedah penyediaan proses yang sangat rumit sehingga sangat mengehadkan aplikasi. Tiada kajian yang dijalankan sehingga ini menggunakan kaedah gabungan baharu untuk mengenal pasti nilai signifikan dan optimum bagi pemboleh ubah yang mempengaruhi sintesis penyahmulsifier. Matlamat penyelidikan ini adalah untuk mensintesis demulsifier untuk emulsi minyak mentah menggunakan bahan berasaskan produk semula jadi dan dengan mengaplikasikan kaedah reka bentuk eksperimen. Demulsifier berasaskan minyak jagung berjaya disintesis melalui tindak balas pemeluwapan minyak jagung dengan dietanolamin dengan kehadiran pemangkin yang digunakan semasa pengasingan melalui emulsi air-dalam-minyak (W/O) menggunakan kaedah tindak balas permukaan (RSM). Parameter sintesis yang signifikan dikenal pasti melalui reka bentuk eksperimen adalah suhu 180 °C, masa dua jam, pemangkin 2.5%, dan dietanolamin kepada minyak jagung (D/C) 1/3, v/v. Demulsifier telah dicirikan oleh analisis FTIR, GC-MS, NMR, LC-QTOF-MS dan IFT. Keberkesanan pemisahan surfaktan telah dikaji menggunakan ujian Sany-glass. Keputusan menunjukkan bahawa produk baru telah menyahmulsi emulsi W/O dengan cekap dengan kadar pemisahan 98% dicapai. Pengaruh masa mendap, dos demulsifier, suhu, dan pH ke atas kecekapan demulsifikasi telah dikaji. Kecekapan pengasingan meningkat dengan peningkatan dalam masa mendap, dos penyahmulsifikasi, dan pH, manakala keadaan suhu mempercepatkan proses penyahmulsifikasi. Ketegangan interfacial berkurangan dengan peningkatan dos demulsifier, dan rancangan komposit utama (CCD) bagi kaedah tindak balas permukaan (RSM) digunakan semasa proses mengoptimumkan penyahmulsifikasian. Proses mengoptimumkan dilakukan menggunakan empat faktor (kandungan air, dos demulsifier, suhu, dan pH). Keadaan optimum untuk demulsifikasi dicapai pada kandungan air 40%, dos 3500 ppm, suhu 60 °C dan pH 8. Kesimpulannya, keputusan pengesahan menunjukkan keputusan yang baik dengan data eksperimen, manakala demulsifier yang baru menunjukkan prestasi cemerlang dan dianggap menepati untuk demulsifikasi emulsi W/O.

ABSTRACT

Water in oil emulsion occurs at many stages in the production and treatment of crude oil. The presence of water causes several operational problems like corrosion in equipment and pipelines. It is also an environmental/health threat, can interfere with refining operations, and generally increases the cost of oil production. Therefore, for economic, environmental, and operational reasons, it is essential to separate the water from the oil before transporting or refining the latter. The most effective method for overcoming this problem is to demulsify the crude using chemical demulsifiers through the synthesis of a demulsifier. Some gaps remain through this process, including that chemical demulsifiers, though effective, are mostly petroleum-based and refractory organic polymers. They often subject the ambient ecosystem to more hazardous risks and use highly complicated preparation methods, greatly limiting their applications. No study has yet been conducted using a new mix design to identify the significant and optimum values of the variables which affect the synthesis of a demulsifier. The aim of this research was to synthesise a demulsifier for crude oil emulsion using materials based on natural products and by applying the design of experiments method. A corn oil–based demulsifier was successfully synthesised through the condensation reaction of corn oil with diethanolamine in the presence of a catalyst applied during separation via a water-in-oil (W/O) emulsion using response surface methodology (RSM). The significant parameters of synthesis, identified through design of experiments, included a temperature of 180 °C, a time of two hours, a 2.5% catalyst, and diethanolamine to corn oil (D/C) 1/3, v/v. The demulsifier was characterised by FTIR, GC-MS, NMR, LC-QTOF-MS, and IFT analyses. The surfactant's separation efficacy was studied using the Sany-glass test. The results showed that the new product efficiently demulsified the W/O emulsion, with 98% separation achieved. The influence of the settling time, demulsifier dosage, temperature, and pH on the demulsification efficiency were investigated. The separation efficiency increased with increases in the settling time, demulsifier dose, and pH, while the temperature conditions accelerated the demulsification process. The interfacial tension decreased with an increased dosage of the demulsifier, and the central composite design (CCD) of the response surface methodology (RSM) was employed during the optimisation demulsification process. The optimisation process was performed using four factors (water content, demulsifier dosage, temperature, and pH). The optimum conditions for demulsification were achieved at water content 40%, dosage 3500 ppm, temperature 60 ºC, and pH 8. Finally, the validation of the results showed good agreement with the experimental data, while the new demulsifier demonstrated excellent performance and was deemed promising for demulsifying W/O emulsions.

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