ISOLATION AND MODIFICATION OF ORGANOSOLV LIGNOCELLULOSIC COMPOUNDS AS DEMULSIFIERS FOR CRUDE OIL DEMULSIFICATION

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ABSTRACT

The most challenging aspect in petroleum industry is the formation of undesired emulsion, which causes major problems such as equipment corrosion, excess cost on transportation as well as low quality oil production. The use of chemical additives of so called demulsifier is widely used along with other conventional methods to accelerate crude oil and brine separation. The demulsifier selection recently moves towards low cost environmental friendly materials. The potential use of lignocellulose based-demulsifier for crude oil demulsification has not been explored yet due to lack of research in this regard. The present study thus, focused on the synthesis of demulsifiers based on lignocellulosic compounds from empty fruit bunch and so are cellulose derivatives as an alternative demulsifier for crude oil demulsification. The oil palm empty fruit bunch was treated by two-stage organosolv treatment and peroxide bleaching to extract lignocellulose compounds. The cellulose derivatives were prepared by modification of extracted cellulose. The synthesized demulsifiers were characterized using the Fourier transform infrared, nuclear magnetic resonance spectroscopy, gel permeation chromatography, thermogravimetric analyzer, scanning electron microscope, X-Ray diffractometer as well as relative solubility number measurement. The performance of demulsifiers was evaluated using bottle test method at 250 to 750 mg L^{-1} demulsifier dosage, temperature at 30 to 90 ^{o}C and pH 4 to 10 of brine. The synthetic emulsion system was prepared by emulsifying 10 ‰ salinity of brine and heptane/toluene mixture (7:3) with 20 weight ratio of resin to asphaltene. The result revealed that the cellulose in ionic liquid, 1-Butyl-3-methylimidazolium chloride showed the best performance among the tested lignocellulose compounds with 98 % of phase separation capacity, η and 0.18 mL h^{-1} of phase separation rate, μ with 750 mg L^{-1} of demulsifier dosage at 70 ^{o}C and neutral pH of brine. The synthesized cellulose derivatives also performed at similar performance on phase separation capacity though their phase separation rate was higher than 0.5 mL h^{-1}. The interfacial tension was reduced from 30 mN m^{-1} to 19 mN m^{-1}. The selected demulsifiers (bleaching filtrate BF-1, quarternized cellulose, QC and cellulose, DC) from each type of lignocellulose based demulsifier were used in the kinetic study. Demulsifier DC and QC followed kinetic controlled adsorption while BF-1 followed diffusion controlled adsorption. For flocculation and coalescence study, water droplets aggregation showed coalescence as controlling step for all demulsifiers DC, QC and BF-1. Finally, the present synthesized demulsifiers may become an alternative chemical for crude oil demulsification due to their promising demulsification performance as biodegradable materials.

ABSTRAK

Aspek yang paling mencabar dalam industri petroleum adalah pembentukan emulsi yang tidak diingini, yang mana menyebabkan masalah-masalah besar seperti kakisan peralatan, lebihan kos pengangkutan dan juga penghasilan minyak berkualiti rendah. Penggunaan bahan kimia tambahan yang dipanggil pemisah emulsi digunakan secara meluas di samping kaedah konvensional lain untuk mempercepatkan pemisahan minyak mentah dan air bergaram. Pemilihan pemisah emulsi baru-baru ini beralih kepada bahan yang berkos rendah dan mesra alam. Penggunaan pemisah emulsi yang berpotensi berasaskan lignoselulosa untuk pemisahan emulsi minyak mentah masih tidak diterokai disebabkan kekurangan penyelidikan dalam perkara ini. Penyelidikan ini dengan itu, tertumpu kepada sintesis pemisah emulsi berasaskan sebatian lignoselulosa daripada tandan kelapa sawit kosong dan juga derivatif selulosa sebagai satu pemisah emulsi alternatif untuk pemisahan emulsi minyak mentah. Tandan kelapa sawit kosong dirawat dengan dua peringkat rawatan pemelarutan organik dan pelunturan peroksida untuk mengekstrak sebatian-sebatian lignoselulosa. Terbitan selulosa telah disediakan melalui pengubahsuaian terhadap selulosa yang diekstrak. Pemisah emulsi yang telah disintesis telah dicirikan menggunakan inframerah transformasi Fourier, spektroskopi resonans magnetik nuklear, kromatografi penelapan gel, penganalisis termogravimetrik, mikroskop elektron imbasan. penganalisis pembelauan X-ray dan pengukuran nombor keterlarutan relatif. Prestasi pemecah emulsi dinilai menggunakan kaedah ujikaji botol pada 250 hingga 750 mg L^{-1} dos pemisah emulsi, suhu 30 ^{o}C hingga 90 ^{o}C dan pH 4 hingga pH 10 air garam. Sistem emulsi yang sintetik telah disediakan dengan mengemulsikan 10 ‰ kemasinan air garam dan campuran heptana/toluena (7:3) dengan ratio berat damar kepada asfaltena 20. Keputusan mendedahkan selulosa di dalam cecair bersifat ionik iaitu 1-Butyl-3-metilimidazolium klorida (BMIMCl) menunjukkan prestasi terbaik antara bahan lignoselulosa yang diuji dengan 98 % kapasiti pemisahan fasa, η and 0.18 mL h^{-1} kadar pemisahan fasa, μ menggunakan 750 mg L^{-1} dos pemisah emulsi pada 70 ^{o}C dan pH air garam yang neutral. Derivatif selulosa yang dihasilkan juga berprestasi pada prestasi yang sama terhadap kapasiti pemisahan fasa, namun kadar pemisahan fasanya lebih tinggi daripada 0.5 mL h^{-1}. Nilai ketegangan pada permukaan diturunkan daripada 30 mN m^{-1} kepada 19 mN m^{-1}. Pemisah emulsi yang dipilih (turasan pelunturan BF-1, sukuan selulosa, QC and selulosa, DC) daripada setiap jenis pemisah emulsi berasaskan selulosa telah digunakan dalam kajian kinetik. Pemisah emulsi DC and QC mematuhi penjerapan yang dikawal oleh kinetik manakala BF-1 mematuhi penjerapan yang dikawal oleh resapan. Bagi kajian penggelompokan dan penyatuan, pengagregatan titisan air menunjukkan penyatuan sebagai langkah kawalan untuk pemisah emulsi DC, QC dan BF-1. Akhirnya, pemisah emulsi yang telah disintesis boleh menjadi bahan kimia alternatif untuk pemisahan emulsi minyak mentah oleh kerana prestasi yang menjanjikan sebagai bahan terbiodegradasi.

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LIST OF ABBREVIATIONS

AFM	-	Atomic force microscopy
AGU	-	Anhydroglucose unit
BF	-	Bleaching filtrate
BL	-	Black liquor
BS	-	Bleaching solution
CHPTAC	-	3-chloro-2-hydroxypropyl trimethyl ammonium chloride
CMC	-	Critical micelle concentration
DS	-	Degree of substitution
EFB	-	Empty fruit bunch
EO/PO	-	Ethylene oxide and propylene oxide
EPPE	-	Epoxypropylphenylether
FTIR	-	Fourier transform infrared
GPC	-	Gel permeation chromatography
HEC	-	Hydroxyethyl cellulose
HLB	-	Hidrophile-lipophile balance
IFT	-	Interfacial tension
LCM	-	Lignocellulosic material
LCP	-	Lignocellulosic polymer
NMR	-	Nuclear magnetic resonance
O/W	-	Oil-in-water
OPB	-	Oil palm biomass
OS	-	Organosolv solution
PHPHEC	-	Phenoxyhydroxypropylhydroxyethyl cellulose
QC	-	Quaternized cellulose
RSN	-	Relative solubility number
SEM	-	Scanning electron microscopy
TGA	-	Thermogravimetric analysis
W/O	-	Water-in-oil
XRD	-	X-ray diffraction

LIST OF SYMBOLS

μ_o	-	Oil phase separation rate (mL h^{-1})
μ_w	-	Water phase separation rate (mL h^{-1})
a_o	-	Flocculation rate ($(cm^3)^{-1}$)
c_s	-	Subsurface concentration
c_o	-	Initial bulk concentration
D	-	Diffusion coefficient (cm^3 s^{-1})
D_s	-	Dosage (mg L^{-1})
K	-	Rate of coalescence (s^{-1})
n_0	-	Number of droplets at time 0
n_t	-	Number of droplets at time t
t	-	Time (h)
V_S	-	Volume of oil/water separated (mL)
V_T	-	Total volume of oil/water (mL)
γ	-	Interfacial tension (Nm m^{-1})
ε	-	Interfacial viscosity (mN.s m^{-1})
η_o	-	Oil phase separation capacity (%)
η_w	-	Water phase separation capacity (%)
τ	-	Integration variable
R^2	-	Coefficient of determination
v/v	-	Volume per volume (mL/mL)
T	-	Temperature (^oC)
Y_i	-	Yield percentage (wt. %)

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Crude oil petroleum consists of a complex mixture of hydrocarbons of various molecular weights that recovered mostly by drilling process from under earth's surface. This presence of water from oil field brine and water injection accidentally forms undesirable water-in-crude oil emulsion where water is dispersed in crude oil. The dispersed water in crude oil is usually known as water cut in oil and gas industry. High turbulence and surface active compounds add to the reasons for the emulsion formation (Abdel-Aal *et al.*, 2003). This stabilized emulsion thus creates several problems such as corrosion of pipelines, pumps, production equipment and downstream overhead distillation column (Abdel-Azim *et al.*, 2010), growth of microorganism in the water-wetted parts of the pipeline and storage tanks, catalyst poisoning and excess expenses for pumping or transporting water via pipeline or tanks (Abdel-Azim *et al.*, 2010; Al-Sabagh *et al.*, 2011c). In order to resolve this problem, several techniques were established but the combination of chemical demulsification together with thermal and mechanical support is employed in crude oil dehydration treatment.

The demulsifiers is vital in dehydration process in addition to the heating and mechanical treatment. They act as emulsion destabilizer by replacing surface active film at the oil-water interface, following with rupture the film and promote water droplet flocculation and coalescence. In practice, demulsifiers used for breaking up water-in-crude oil emulsion are typically based on several chemistries such as alcohol, phenols, alcohol/amines, resin (Peña *et al.*, 2005), nonylphenols (Fan *et al.*, 2009), polyhydric alcohols and sulphonic salts, polymeric surfactants of ethylene oxide/propylene oxide (EO/PO) copolymer (Kelland, 2009; Kokal, 2005).

However, these demulsifiers are mainly synthesized from non-natural petroleum sources, creating an environmental issue. To meet ever-increasing stringent environmental regulations, natural biodegradable demulsifiers were introduced. For example, rhamnolipid (Long *et al.*, 2013), fungal spore (Vallejo-Cardona *et al.*, 2017), chitosan derivatives (Bratskaya *et al.*, 2006) and cellulose derivatives (Feng *et al.*, 2009; Roostaie *et al.*, 2017) were applied as demulsifier in various petroleum emulsion.

There were much attention on oil palm biomass (OPB) as an alternative source for economic utilization in electricity, biofuels, fertilizer and other high-value added products (Onoja *et al.*, 2018). Malaysia as one of the main palm oil producers was forecasted to produce 85 to 111 million tonnes of biomass from oil palm industry including oil palm plantation and processing (Basiron, 2007). Empty fruit bunches (EFB) in particular is a versatile oil palm biomass by producing renewable product as biofuel, organic compost, palm fibers, bio-degradable products as well as for life science applications such as for extraction of lignocelluloses and bio-sugar. However, this biomass is largely unutilized and causes severe environmental problems, which explains why there have currently gained considerable interests towards utilization of this biomass, supported by several initiatives of the Malaysian government.

Lignocellulosic polymers (LCPs) so called cellulose, hemicellulose and lignin are the main polymer component in the plant cell wall. These natural polymers are fractionated which could be potentially applied as demulsifier considering its surfactant properties. Lignocellulose based polymer was already applied as a kind of cheaper and biodegradable product in medical products, protective colloids, coating, surfactants, hair conditioners, antistatic agents, dispersion agents, adhesives and textiles (Varshney and Naithani, 2011). The first cellulose based demulsifier, ethyl cellulose was introduced by Feng *et al.* (2009) in demulsifying bitumen-in-water emulsion while hemicellulose and lignin had not been studied as demulsifier yet. Thus, the lignocellulosic polymers of EFBs biomass are likely having a big prospect as demulsifier to resolve crude oil emulsion in the petroleum industry.

1.2 Problem Statements

Malaysia's palm oil biomasses (OPB) are produced in abundance. For instance, in one tonne of oil production, the total oil palm biomass generated from palm oil industry is 2.3 tonnes including trunks, fronds, kernels, shells and empty fruit bunches (Chang, 2014). The 14.1 million tonnes Malaysia palm oil production in 2017 makes it verging on 32.43 million tonnes OPBs. From that amount, almost half of it is empty fruit bunches (EFB). The EFBs went into wealth transformation, some of which are electricity generation, mulch for plantation (Menon *et al.*, 2003), conversion into pulp and paper (Bajpai, 2012) and production of roughage for animal feeds (Atil, 2004), and bio-fuel production (Chang, 2014). Even though, it is still incomparable with the large population of EFBs biomass, which should be immensely exploited to reduce biomass waste and create sustainability in industrial field.

The EFBs biomass has vastly potential for industrial application not only because of abundant supply, but also because of its high composition of lignocelluloses polymer compounds namely cellulose, hemicelluloses and lignin (Law *et al.*, 2007). The natural polymers consisted in EFBs has potential as demulsifier for crude oil emulsion. LCPs based materials have very wide applications in industries concerned with oilfield treatments, medical products, protective colloids, coating, surfactants, hair conditioners, antistatic agents, dispersion agents, adhesives and textiles (Dumitriu, 2000; Hon, 1996). Another potential application of these polymers which has not been fully explored is demulsifier for crude oil emulsion. Established demulsifier such as formaldehyde resins, alkoxylated amines, or mixtures of them have issue on their biodegradability, toxicity and cost (Guzmán-Lucero *et al.*, 2010; Issaka *et al.*, 2015). Thus, searching for more environmental friendly, toxicologically harmless, cheap and easily available demulsifiers became main concern in petroleum industry.

Unlike hemicellulose and lignin that can be dissolved in either water or general solvents, cellulose has highly order hydrogen bonding network and high crystallinity which both detract cellulose solubility. However, the unique structure and reactivity of cellulose thus provide various possibilities for the design and development of cellulose based material through the chemical modifications. Recently, ethylcellulose (EC) was found effective in dewatering water-in-dilute bitumen emulsion (Feng *et al.*, 2009) though it was synthetic cellulose derivatives. Therefore, naturally lignocelluloses polymer based materials from EFBs biomass were used as demulsifiers for crude oil demulsification and their potential was demonstrated in this study.

1.3 Objectives and Scopes

In order to resolve the problems discussed in Section 1.2, four consecutive objectives and scopes were featured in this research:

a) To isolate and characterize lignocellulosic compounds from empty fruit bunches.

Cellulose, hemicelluloses and lignin polymers were extracted from EFB by using two stages treatment (organosolv treatment and hydrogen peroxide bleaching treatment). The EFB was characterized by proximate analysis (ash, moisture and acid insoluble lignin content and NaOH and water solubility). The isolated LCPs were characterized by scanning electron microscopy (SEM), X-ray diffraction analysis (XRD), Fourier transform infrared (FTIR), nuclear magnetic resonance (NMR) spectroscopy, gel permeation chromatography (GPC) and thermogravimetric analysis (TGA). The process will create several impacts such as precipitation.

b) To evaluate organosolv filtrates from EFB treatment and lignocellulose compounds extracted from EFB treatment as demulsifiers for crude oil demulsification.

Four filtrates yielded from organosolv treatment of EFB before lignin and hemicellulose precipitation were tested as demulsifier. In the second part, isolated lignocellulosic compounds (i.e. cellulose, hemicellulose and lignin) were dissolved in 1-butyl-3-methylimidazolium chloride before evaluated. The performance of organosolv filtrates, lignocellulosics compounds and cellulose derivatives as the demulsifiers was measured by using bottle test experiment. The bottle test experiment was set up and phase separation capacity and rate were determined. The selected demulsifiers from these categories were furthered evaluated at various experimental conditions: temperature (30 ^{o}C -90^{o}C), demulsifier dosage (200 mg L^{-1} to 1000 mg L^{-1}) and initial brine pH (pH 3 to pH 9). The performance was also evaluated by analyses such as brine and crude oil residue in separated crude oil and brine, interfacial tension and interfacial viscosity.

c) To synthesize and evaluate cellulose derivatives as demulsifiers for crude oil demulsification.

Lastly, cellulose was selected to be modified as cellulose derivatives as demulsifiers. The isolated cellulose from EFB was modified by quaternization and etherification process. Then, synthesized ether cellulose was hydrophobically modified. The characterizations was done to these synthesized cellulose based demulsifiers involving Fourier transform infrared (FTIR), nuclear magnetic resonance spectroscopy (NMR) spectroscopy and gas permeation chromatography (GPC). Syntesized cellulose derivatives were dissolved in toluene as their solvent. The cellulose derivatives demulsifiers undergo same experimental procedures as demulsification test for organosolv filtrates and lignocellulose compound.

d) To study the crude oil demulsification process using organosolv filtrates, lignocellulosics compounds and cellulose derivatives as demulsifiers

The demulsification process was studied for demulsifier adsorption and water droplet coagulation process. For demulsifier adsorption process, dynamic interfacial tension data were analyzed using the existing kinetic model (diffusion controlled adsorption at liquid-liquid interface). For water droplets coagulation, number of water droplets data obtained by stereomicroscope was analyzed using several kinetic models (i.e. Borwankar, Azizi and Nikazar and Pramudono). The model analyses were discussed towards understanding the mechanism of the demulsification process.

1.5 Thesis Outline

This thesis contains 5 chapters in overall. Chapter 1 presents research background, problem statement, objectives and scopes of the research, thesis outline and chapter summary. This chapter describes the motivation behind using proposed biodegradable demulsifiers in crude oil emulsion from the background of study's field. The literature review is presented in Chapter 2 to support the present work. The past researches related to crude oil emulsion problem in oil and gas industry, demulsification methods used in water-oil separation, LCPs as potential demulsifier and technical aspect of demulsification process were critically reviewed.

Chapter 3 discussed about the research methodology, which includes materials and experimental and analytical procedures that had been used to achieve research objectives. The experimental works include synthesis, characterization and crude oil demulsification measurement. The research findings of demulsifier synthesis and characterization, crude oil demulsification performance and evaluation of crude oil demulsification process were presented and discussed specifically in Chapter 4. Chapter 5 presents the conclusions of the study and recommendations for future studies. Following it, the list of references cited in the thesis was listed.

1.6 Summary

The abundant source of oil palm empty fruit bunch (EFB) is appealing in the view point of its economic and environmental value. Considering the existence of nonbiodegrable, high toxic and expensive demulsifier being used in demulsifying crude oil emulsion, lignocelulosic polymers (LCPs) contained in EFB, cellulose in particular may offers a solution regarding this petroleum treatment process. Thus, it is necessary to study the performance of new lignocellulose based biodegradable demulsifier from EFB as an alternative low cost, green and non-toxic material as well as to contribute to the sustainability of the environment through utilization of EFB crop waste.

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APPENDIX A

ISOLATION OF LIGNOCELLULOSE COMPOUNDS FROM EFB

Appendix A.1 Data Proximate Analysis of EFB

Analysis	1st	2nd	3rd	AVG (%)	STDEV (%)
Moisture content	9.12	9.560	10.359	9.70	0.69
Extractives	4.384	3.478	3.114	3.66	0.84
Matter soluble in caustic soda	33.772	32.861	35.202	33.77	1.87
Cold water solubility	8.530	9.491	10.135	8.53	1.14
Hot water solubility	5.894	5.192	5.650	5.89	0.58
Ash content	2.630	1.848	2.282	2.24	2.08

Appendix A.2 Data Yield Percentage of Lignocellulose Polymers (LCPs) from EFB

Lignocellulose Polymers	1st	2nd	3rd	Average	STDEV (%)
Cellulose	44.23	44.42	46.55	45.07	1.29
Hemicellulose 1	3.99	7.66	7.98	6.54	2.22
Hemicellulose 2	19.15	12.12	12.63	14.63	3.92
Lignin 1	12.23	8.25	8.50	9.66	2.23
Lignin 2	3.47	5.43	4.99	4.63	1.03

APPENDIX B

DEMULSIFIER PERFORMANCE

Appendix B.1 Data phase separation capacity of OPFs with experimental condition: D_s = 250 mg L^{-1}, temperature = 50 ^oC, brine pH= 6.6 for 20 hours

	Phase separation capacity, η (%)	
	Water separation	Oil separation
OS	14	12
BL-1	2	20
BL-2	34	48
BS	1	10
BF-1	26	20
BF-2	40	40
D0	3	14

Appendix B.2 Data effect of temperature on phase separation capacity of OPFs with experimental condition: D_s = 250 mg L^{-1}, brine pH= 6.6 for 20 hours

	Phase separation capacity, η (%)							
	Water separation				Oil separation			
T (^oC)	30	50	70	90	30	50	70	90
BL-2	40	100	100	100	48	100	100	100
BF-1	30	60	78	94	50	54	60	76
BF-2	56	100	100	100	48	100	100	100
D0	1	20	30	70	36	40	70	80

Appendix B.3 Data effect of brine pH on phase separation capacity of OPFs with experimental condition: D_s = 250 mg L^{-1}, temperature = 50 ^oC for 20 hours

	Phase separation capacity, η (%)							
	Water separation				Oil separation			
pH	3.0	6.6	7.0	9.0	3.0	6.6	7.0	9.0
BL-2	24	100	100	48	28	100	98	30
BF-1	28	54	62	14	32	60	82	44
BF-2	60	100	100	46	62	100	98	54
D0	0.05	14	10	0.05	4	10	12	1

Appendix B.4 Data effect of demulsifier dosage on phase separation capacity of OPFs with experimental condition: temperature = 50 ^oC, brine pH= 6.6 for 20 h

	Phase separation capacity, η (%)					
	Water separation			Oil separation		
Ds (mg L^{-1})	250	500	750	250	500	750
BL-2	68	77	100	71	88	100
BF-1	18	34	76	30	54	86
BF-2	54	88	100	60	72	100

Appendix B.5 Data phase separation capacity of LCPs with experimental condition: D_s = 250 mg L^{-1}, temperature = 50 ^oC, brine pH= 6.6 for 20 h

	Phase separation capacity, η (%)	
	Water separation	Oil separation
D_C	80	82
D_L	25	36
D_H	1	2
D_{IL}	20	20
D_0	3	3

Appendix B.6 Data effect of temperature on phase separation capacity of LCPs with experimental condition: D_s = 250 mg L^{-1}, brine pH= 6.6 for 20 h

	Phase separation capacity, η (%)							
	Water separation				Oil separation			
T (^oC)	30	50	70	90	30	50	70	90
D_C	80	90	100	100	90	92	100	100
D_L	20	66	88	90	34	72	94	94
D_0	20	33	48	70	24	40	54	77

Appendix B.7 Data effect of brine pH on phase separation capacity of LCPs with experimental condition: D_s = 250 mg L^{-1}, temperature = 50 ^oC for 20 h

	Phase separation capacity, η (%)							
	Water separation				Oil separation			
pH	3	6.6	7	9	3	6.6	7	9
D_C	48	90	94	53	52	90	92	60
D_L	32	64	58	26	42	70	66	33
D_0	1	33	28	1	4	38	30	5

Appendix B.8 Data effect of demulsifier dosage on phase separation capacity of LCPs with experimental condition: temperature = 50 ^oC, brine pH= 6.6 for 20 h

	Phase separation capacity, η (%)					
	Water separation			Oil separation		
D_s (mg L^{-1})	250	500	750	250	500	750
D_C	80	84	90	85	88	92
D_L	24	33	62	30	34	68

Appendix B.9 Data phase separation capacity of cellulose derivatives with experimental condition : D_s = 250 mg L^{-1}, temperature = 50 ^oC, brine pH = 6.6 for 20 h

	Phase separation capacity, η (%)	
	Water separation	Oil separation
QC	100	100
HC	98	96
PC	90	96
D0	10	32

Appendix B.10 Data temperature effect on phase separation capacity of cellulose derivatives with experimental condition: D_s = 250 mg L^{-1}, brine pH= 6.6 for 20 h

	Phase separation capacity, η (%)							
	Water separation				Oil separation			
T (^oC)	30	50	70	90	30	50	70	90
QC	100	100	100	100	100	100	100	100
HC	98	100	100	100	98	100	100	100
PC	96	100	100	100	96	100	100	100
D0	20	33	48	70	24	40	54	77

Appendix B.11 Data brine pH effect on phase separation capacity of cellulose derivatives with experimental condition: D_s = 250 mg L^{-1}, temperature = 50^oC for 20 h

	Phase separation capacity, η (%)							
	Water separation				Oil separation			
pH	3	6.6	7	9	3	6.6	7	9
QC	60	100	100	40	56	100	100	40
HC	20	100	100	24	22	100	100	20
PC	20	98	100	60	18	98	100	30
D0	1	33	28	1	4	38	30	5

Appendix B.12 Data demulsifier dosage effect on phase separation capacity of cellulose derivatives with experimental condition: temperature = 50°C, brine pH= 6.6 for 20 h

	Phase separation capacity, η (%)							
	Water separation				Oil separation			
D_s (mg L^{-1})	250	500	750	1000	250	500	750	1000
QC	40	60	100	100	46	64	98	100
HC	20	52	98	100	30	58	96	100
PC	20	60	100	100	30	74	100	100

Appendix B.13 Data molar ratio effect on phase separation capacity of cellulose derivatives with experimental condition: D_s = 250 mg L^{-1}, temperature = 50°C, brine pH= 6.6 for 20 h

	Phase separation capacity, η (%)					
	Water separation			Oil separation		
Molar ratio	1:06	1:09	1:12	1:06	1:09	1:12
QC	68	100	100	72	96	100
HC	90	98	100	90	98	96
PC	70	100	100	66	100	100

Appendix B.14 Data phase separation rate of OPFs with experimental condition : D_s = 250 mg L^{-1}, temperature = 50°C, brine pH= 6.6 for 20 h

	Phase separation rate, μ (mL h^{-1})	
	Water separation	Oil separation
OS	0.078	0.062
BL-1	0.012	0.074
BL-2	0.111	0.181
BS	0.004	0.021
BF-1	0.089	0.068
BF-2	0.153	0.171
D0	0.008	0.042

Appendix B.15 Data temperature effect on phase separation rate of OPFs with experimental condition: D_s = 250 mg L^{-1}, brine pH= 6.6 for 20 h

	Phase separation rate, μ (mL h^{-1})							
	Water separation				Oil separation			
T (°C)	30	50	70	90	30	50	70	90
BL-2	0.138	1.008	1.958	2.012	0.414	0.916	1.522	1.557
BF-1	0.132	0.316	0.544	0.655	0.381	0.384	0.436	0.453
BF-2	0.371	0.827	0.932	2.051	0.756	0.861	0.828	2.320
D0	0.003	0.052	0.074	0.191	0.254	0.261	0.322	0.334

Appendix B.16 Data brine pH effect on phase separation rate of OPFs with experimental condition: D_s = 250 mg L^{-1}, temperature = 50°C for 20 h

	Phase separation rate, μ (mL h^{-1})							
	Water separation				Oil separation			
pH	3	6.6	7	9	3	6.6	7	9
BL-2	0.140	1.088	0.903	0.320	0.208	0.791	0.709	0.211
BF-1	0.149	0.354	0.457	0.061	0.258	0.305	0.392	0.311
BF-2	0.402	0.800	0.914	0.338	0.434	0.474	0.599	0.342
D0	0.006	0.081	0.013	0.006	0.035	0.091	0.071	0.012

Appendix B.17 Data demulsifier dosage effect on phase separation rate of OPFs with experimental condition: temperature = 50°C, brine pH= 6.6 for 20 h

	Phase separation rate, μ (mL h^{-1})					
	Water separation			Oil separation		
D_s (mg L^{-1})	250	500	750	250	500	750
BL-2	0.229	0.403	0.611	0.297	0.368	0.426
BF-1	0.014	0.054	0.084	0.053	0.193	0.211
BF-2	0.146	0.292	0.768	0.208	0.313	0.561

Appendix B.18 Data phase separation rate of LCPs with experimental condition: D_s = 250 mg L^{-1}, temperature = 50°C, brine pH= 6.6 for 20 h

	Phase separation rate, μ (mL h^{-1})	
	Water separation	Oil separation
D_C	0.184	0.168
D_L	0.091	0.155
D_H	0.000	0.011
D_{IL}	0.041	0.041
D_0	0.011	0.011

Appendix B.19 Data temperature effect on phase separation rate of LCPs with experimental condition: D_s = 250 mg L^{-1}, brine pH= 6.6 for 20 h

	Phase separation rate, μ (mL h^{-1})							
	Water separation				Water separation			
T (°C)	30	50	70	90	30	50	70	90
D_C	0.205	0.374	0.585	0.834	0.364	0.470	0.628	0.641
D_L	0.062	0.213	0.563	0.682	0.296	0.300	0.676	0.715
D_0	0.116	0.135	0.242	0.317	0.172	0.227	0.321	0.350

Appendix B.20 Data brine pH effect on phase separation rate of LCPs with experimental condition: D_s = 250 mg L^{-1}, temperature = 50°C for 20 h

	Phase separation rate, μ (mL h^{-1})							
	Water separation				Water separation			
pH	3	6.6	7	9	3	6.6	7	9
D_C	0.297	0.374	0.438	0.314	0.343	0.470	0.555	0.380
D_L	0.237	0.213	0.297	0.123	0.277	0.264	0.293	0.246
D_0	0.006	0.135	0.131	0.006	0.018	0.227	0.273	0.018

Appendix B.21 Data demulsifier dosage effect on phase separation rate of LCPs with experimental condition: temperature = 50°C, brine pH= 6.6 for 20 h

	Phase separation rate, μ (mL h^{-1})					
	Water separation			Water separation		
D_s (mg L^{-1})	250	500	750	250	500	750
D_C	0.112	0.170	0.342	0.161	0.205	0.414
D_L	0.057	0.084	0.142	0.066	0.076	0.138

Appendix B.22 Data phase separation rate of cellulose derivatives with experimental condition: D_s = 250 mg L^{-1}, temperature = 50°C, brine pH= 6.6 for 20 h

	Phase separation rate, μ (mL h^{-1})	
	Water separation	Oil separation
QC	0.417	0.329
HC	0.488	0.400
PC	0.388	0.345
D0	0.011	0.011

Appendix B.23 Data temperature effect on phase separation rate of cellulose derivatives with experimental condition: D_s = 250 mg L^{-1}, brine pH= 6.6 for 20 h

	Phase separation rate, μ (mL h^{-1})							
	Water separation				Oil separation			
T (°C)	30	50	70	90	30	50	70	90
QC	0.405	0.530	0.880	1.030	0.431	0.540	0.910	1.100
HC	0.472	0.680	0.940	1.200	0.485	0.630	0.970	1.220
PC	0.390	0.460	0.530	0.860	0.401	0.490	0.590	0.900
D0	0.116	0.135	0.242	0.317	0.172	0.227	0.321	0.350

Appendix B.24 Data brine pH effect on phase separation rate of cellulose derivatives with experimental condition: D_s = 250 mg L^{-1}, temperature = 50^{o}C for 20 h

	Phase separation rate, μ (mL h^{-1})							
	Water separation				Oil separation			
pH	3	6.6	7	9	3	6.6	7	9
QC	0.190	0.530	0.540	0.176	0.210	0.540	0.560	0.198
HC	0.170	0.680	0.649	0.220	0.190	0.630	0.680	0.250
PC	0.082	0.460	0.550	0.200	0.090	0.490	0.530	0.179
D0	0.006	0.135	0.131	0.006	0.018	0.227	0.273	0.018

Appendix B.25 Data demulsifier dosage effect on phase separation rate of cellulose derivatives with experimental condition: temperature = 50^{o}C, brine pH= 6.6 for 20 h

	Phase separation rate, μ (mL h^{-1})							
	Water separation				Oil separation			
D_s (mg L^{-1})	250	500	750	1000	250	500	750	1000
QC	0.176	0.296	0.318	0.530	0.202	0.310	0.369	0.540
HC	0.210	0.330	0.540	0.680	0.230	0.310	0.550	0.630
PC	0.082	0.190	0.290	0.460	0.101	0.179	0.240	0.490

Appendix B.26 Data molar ratio effect on phase separation rate of cellulose derivatives with experimental condition: D_s = 250 mg L^{-1}, temperature = 50^{o}C, brine pH= 6.6 for 20 h

	Phase separation rate, μ (mL h^{-1})					
	Water separation			Oil separation		
Molar ratio	1:06	1:09	1:12	1:06	1:09	1:12
QC	0.070	0.528	0.640	0.084	0.572	0.680
HC	0.145	0.645	0.650	0.170	0.670	0.670
PC	0.153	0.476	0.560	0.200	0.484	0.580

Appendix B.27 Comparative study of selected demulsifiers with experimental condition: D_s = 750 mg L^{-1}, temperature = 50°C, brine pH= 6.6 for 20 h

	Phase separation rate, μ (mL h^{-1})		Phase separation capacity, η (%)	
	Water separation	Oil separation	Water separation	Oil separation
BL-2	1.008	0.916	100	100
BF-1	0.316	0.384	60	54
BF-2	0.827	0.861	100	100
DC	0.374	0.470	90	92
DL	0.213	0.300	66	72
QC	0.303	0.245	100	100
HC	0.680	0.535	100	100
PC	0.562	0.245	100	100

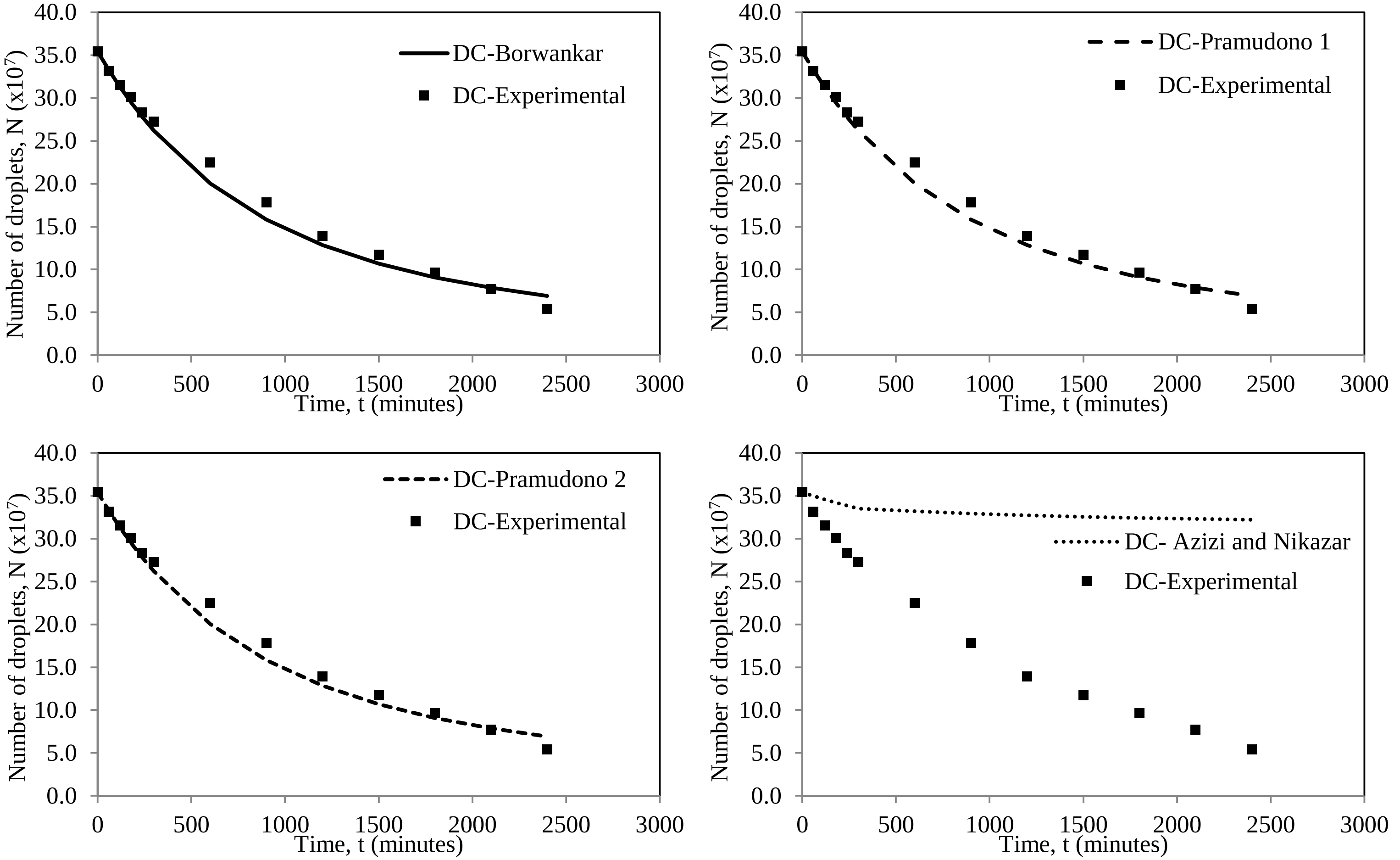
APPENDIX C

KINETIC OF CRUDE OIL DEMULSIFICATION

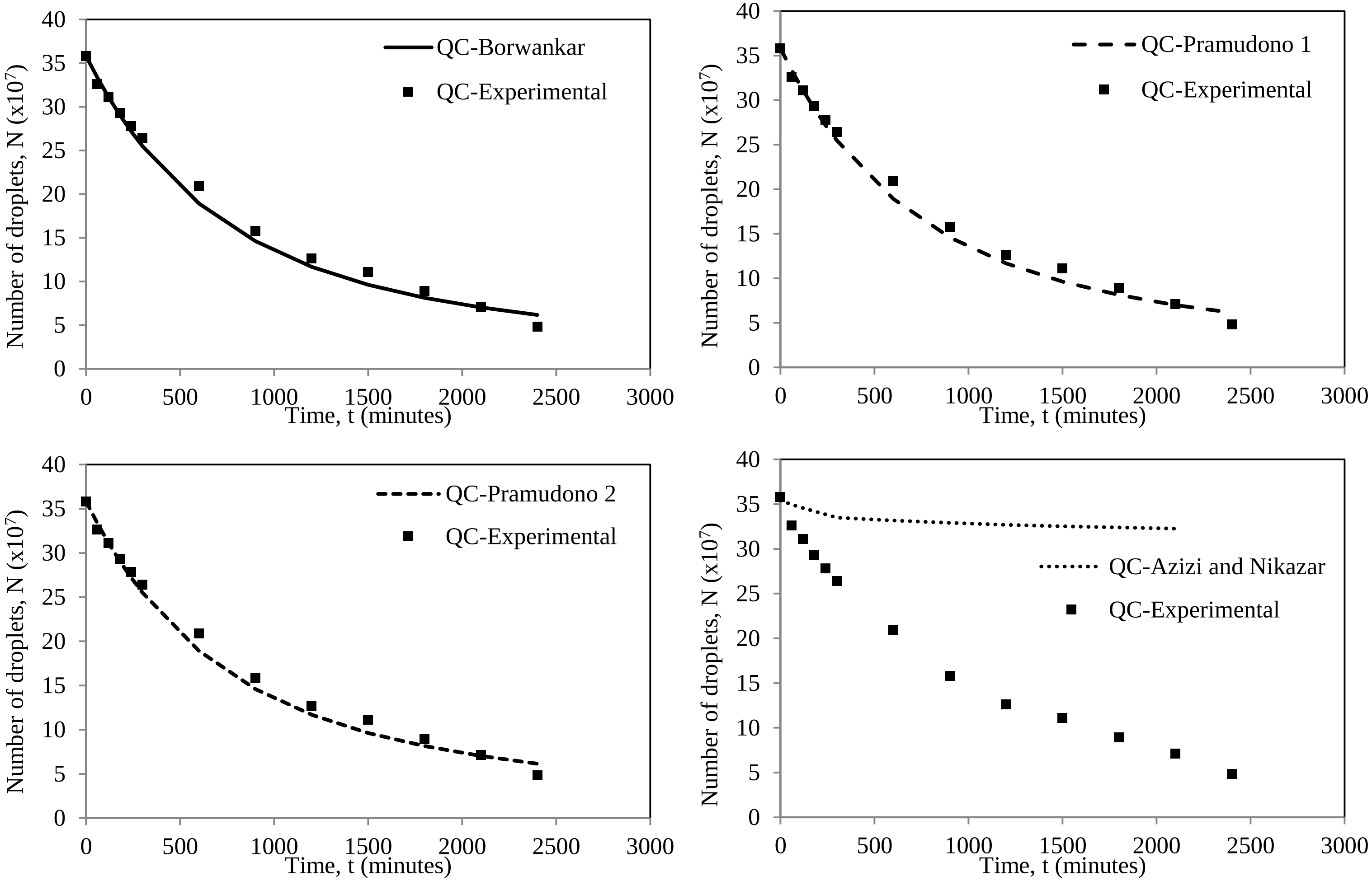
Appendix C.1 Data number of droplets for flocculation and coalescence kinetic of crude oil demulsification process by DC, QC and BL-2 demulsifier

	Number of droplets		
Time (s)	DC	QC	BF-1
0	3540000000	3580000000	3580000000
60	3310000000	3260000000	3410000000
120	3150000000	3110000000	3250000000
180	3010000000	2930000000	3170000000
240	2830000000	2780000000	2980000000
300	2720000000	2640000000	2820000000
600	2250000000	2090000000	2410000000
900	1780000000	1580000000	1980000000
1200	1390000000	1260000000	1420000000
1500	1170000000	1110000000	1230000000
1800	960000000	890000000	1060000000
2100	770000000	710000000	870000000
2400	540000000	480000000	640000000

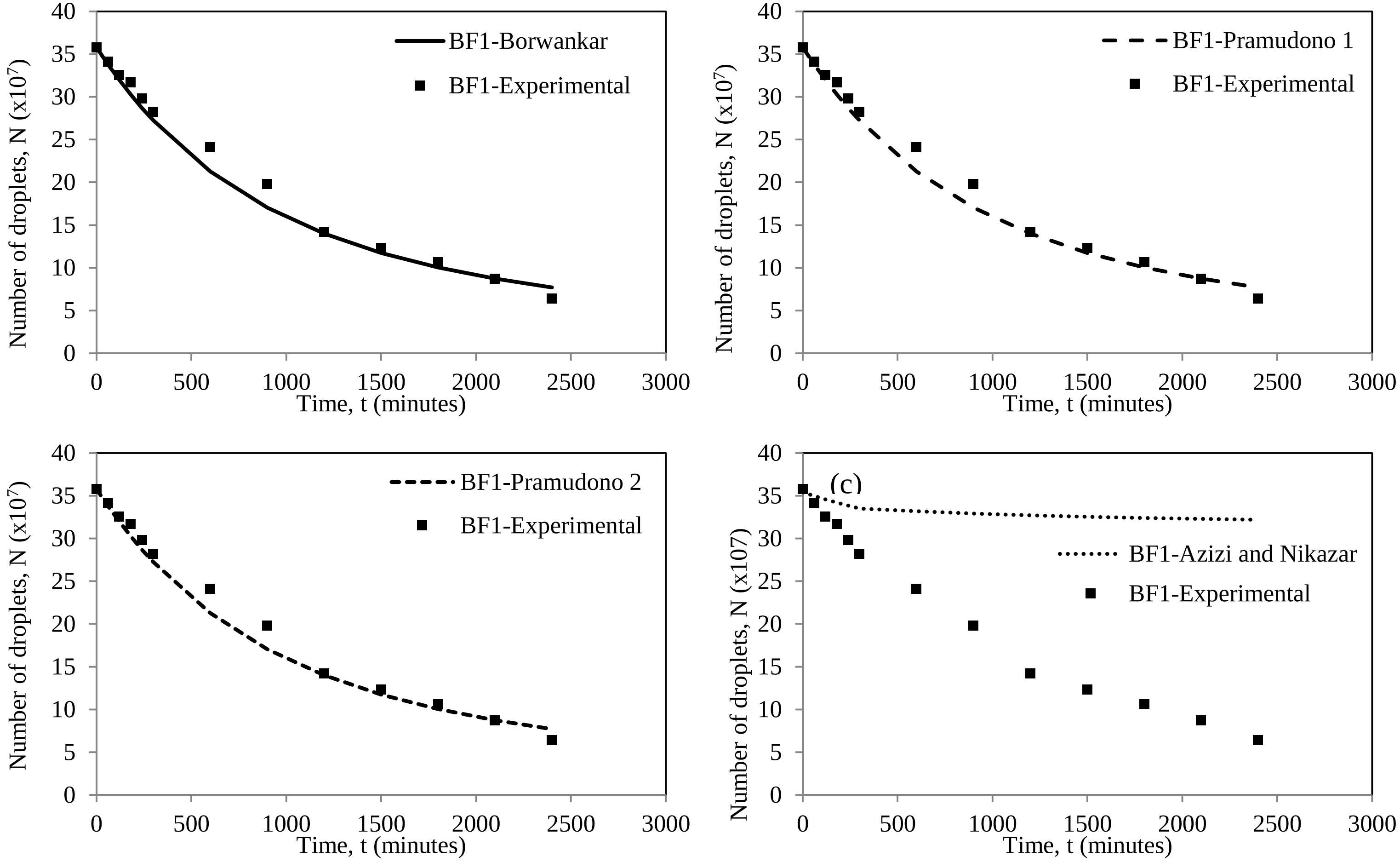
Appendix C.2 Model fitted graph for equilibrium data of number of droplets using demulsifier DC



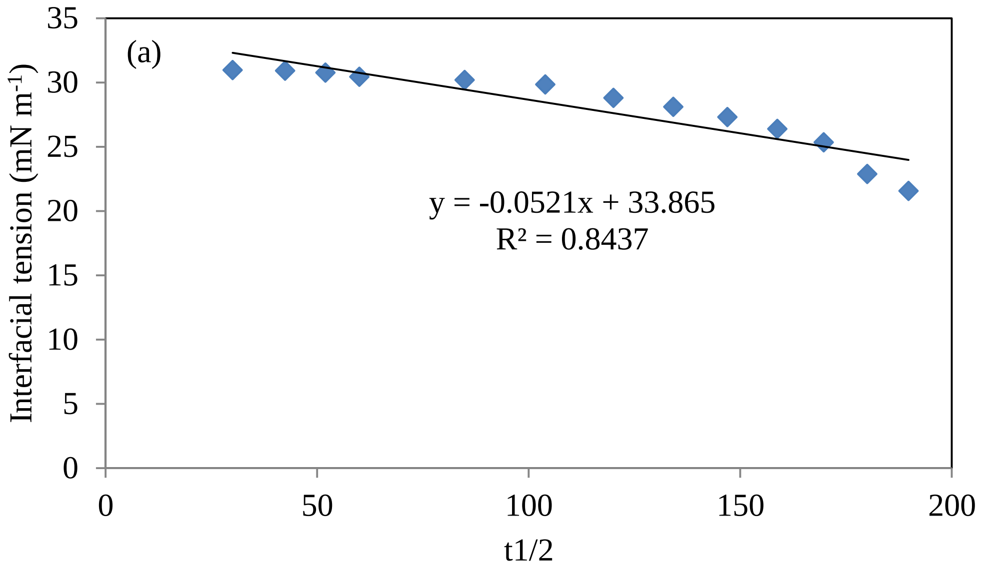
Appendix C.3 Model fitted graph for equilibrium data of number of droplets using demulsifier QC

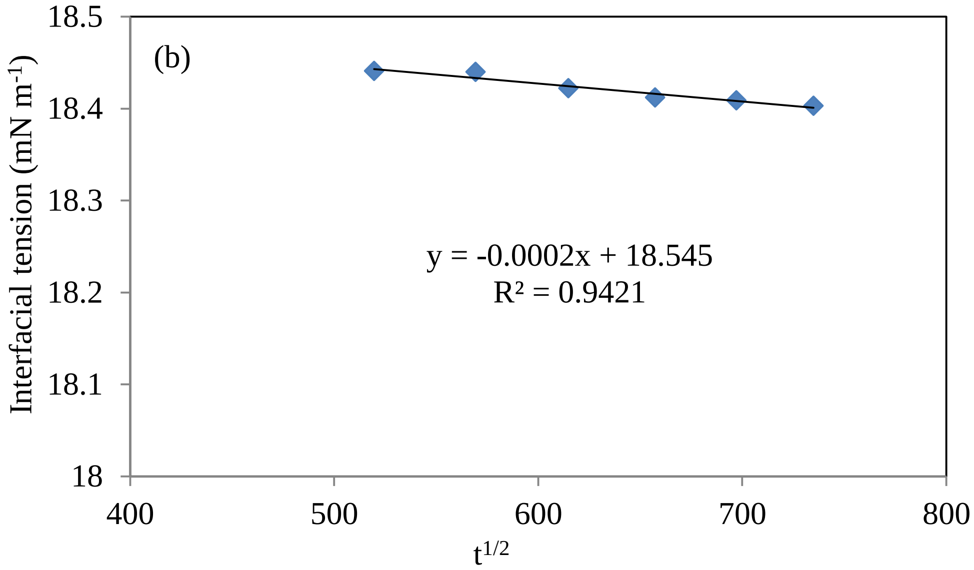


Appendix C.4 Model fitted graph for equilibrium data of number of droplets using demulsifier BF-1

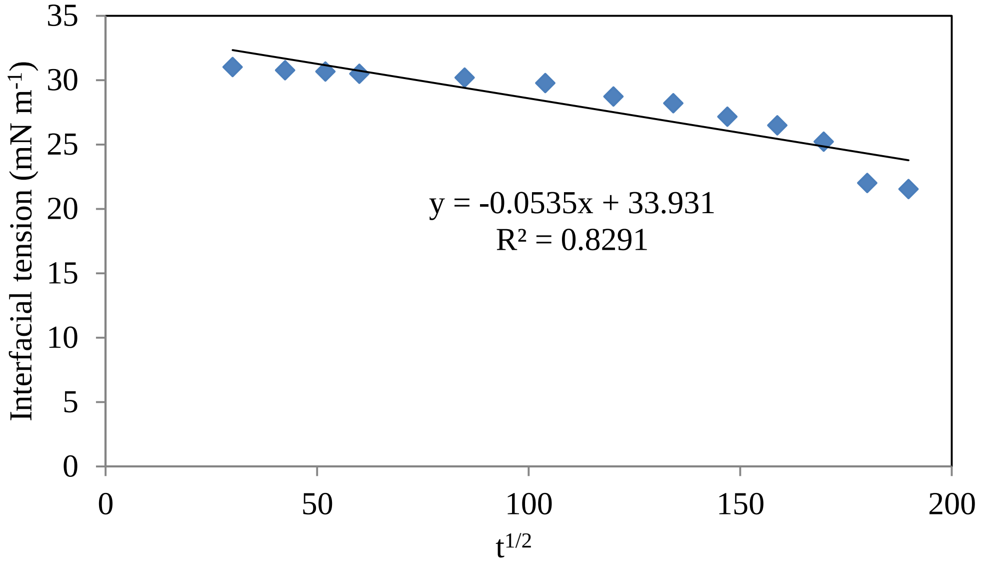


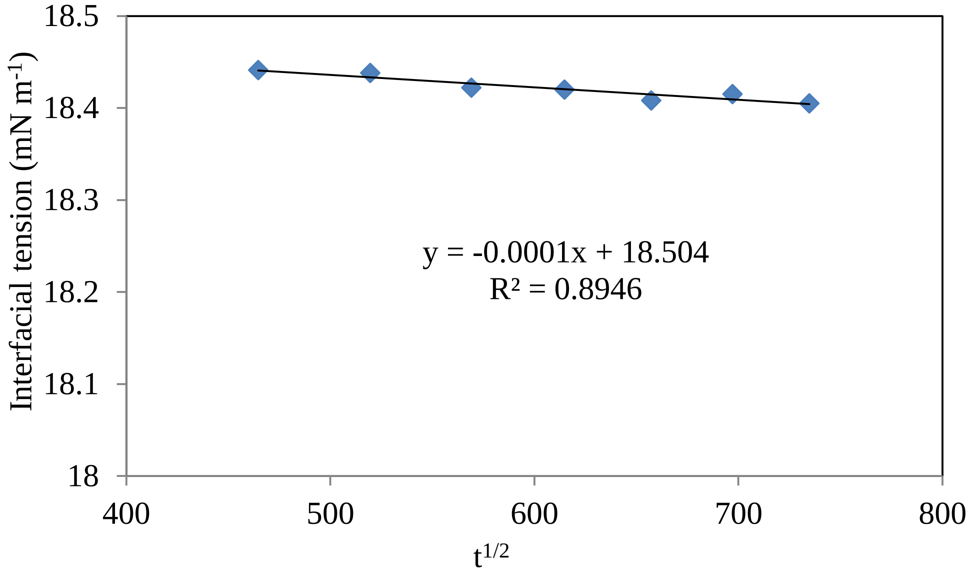
Appendix C.5 Model fitted graph for equilibrium data of interfacial tension using demulsifier BF-1: (a) short time limit and (b) long time limit



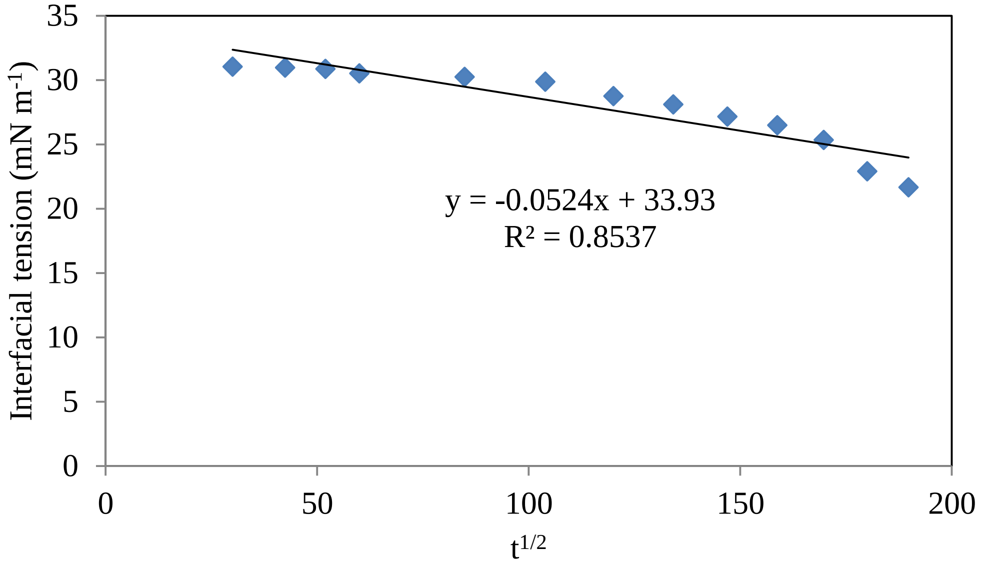


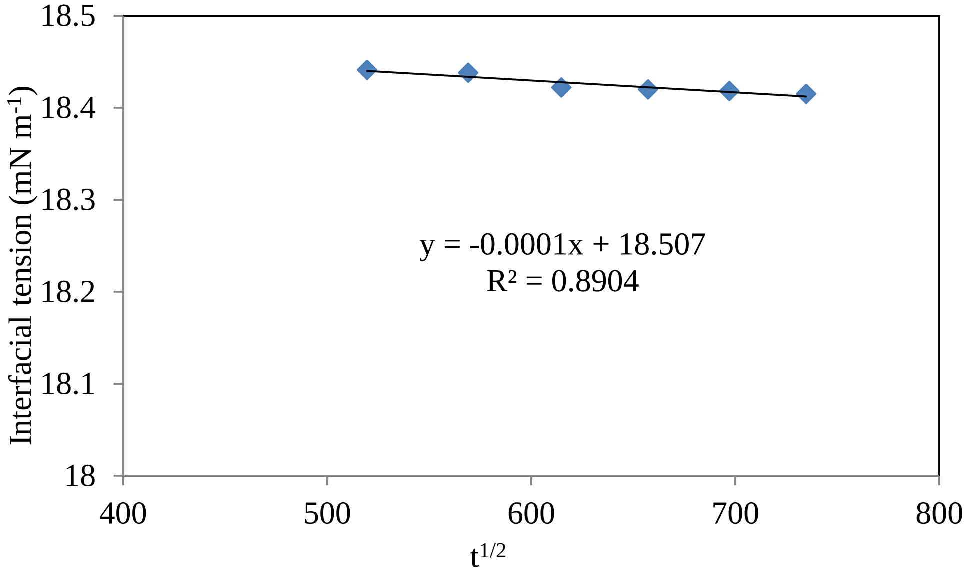
Appendix C.6 Model fitted graph for equilibrium data of interfacial tension using demulsifier QC: (a) short time limit and (b) long time limit





Appendix C.7 Model fitted graph for equilibrium data of interfacial tension using demulsifier BF-1: (a) short time limit and (b) long time limit





LIST OF PUBLICATIONS

Indexed Journal

1. **Tengku Malim Busu, T. N. Z.**, Saman, N., Mohtar, S. S., Md Noor, A. M., Hassan, O., Ali, N., & Mat, H. (2019). An evaluation of lignocellulosic solutions from OPEFB pulping process as demulsifiers for crude oil emulsion demulsification. *Petroleum Science and Technology*, *37*(14), 1675–1682. https://doi.org/10.1080/10916466.2019.1602639. **(Indexed by SCOPUS)**