

# The role of fatty acid of Morinda citrifolia oil as surface- active chemicals on the deinking process of waste paper

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## The role of fatty acid of *Morinda citrifolia* oil as surface-active chemicals on the deinking process of waste paper



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### ABSTRACT

The natural surfactant from *Morinda citrifolia* seed oil was developed for deinking flotation. In this study, the comparative performances of natural surfactant and synthetic surfactant (SS) was investigated. Natural surfactants consist of several compounds element, namely saturated fatty acids, monounsaturated fatty acids, and polyunsaturated fatty acids. The results provide a new phenomenon in which intermolecular bonding occurs between the tail of the carbon chains methyl ester (FAMC) and the ink particles, this is due to the presence of unsaturated compounds (C=C) in the carbon chains structure of fatty acid methyl ester. The results also show that the binding force between synthetic surfactant compounds and ink particles produces a higher viscosity when compared to the bonding forces between FAMC compounds and ink particles. These results indicate that natural FAMC surfactants weaken the molecular bonds of the ink and increase the reactivity of ink particles. These results indicate that the deinking performance of natural surfactants is better than synthetic surfactants.

### 1. Introduction

The flotation process is a significant thing needed in the process of recycling used paper by lifting the ink on the paper. This has been done in Europe, North America, and many other countries. Moreover, for economic and ecological reasons, recovered waste paper has become the main ingredient in producing graphic papers, hygienic papers, and light board topline [1,2]. However, when compared to other ink removal processes such as washing and centrifugal cleaning, flotation deinking is more profitable because the process of purifying water during the recovery process is easier. In fact, this process manages to increase brightness by removing and cleaning dirt stains [3]. On the other hand, to increase its efficiency, fluid dynamics (hydrodynamic) and surface reactions are important factors that must be taken into account during the deinking process [4–6]. Some of the important parameters that occur during the deinking flotation process are the size and shape of the bubbles, the displacement, and interactions between bubbles, agglomeration, and collisions between bubbles in the flotation column [7,8]. Furthermore, the process of removing ink from paper through crumbled worn pulp needs synthetic and automatic states, and the effect of the mechanical force makes particles separate from paper and this is greatly influenced by the hydrodynamic flow of the liquid phase in the pulper and swelling of the paper. On the other hand, research on chemically deinking processes [9,10] and enzymatic deinking for non-impact

printed and photocopy paper [11] has been carried out. Even deinking processes as new and old newspapers [12], and for ray-printed waste paper [13]. Furthermore, researchers have used anionic surfactants to remove photocopy ink on paper as well as on laser prints [14], including pure cationic and nonionic surfactants [15,16,17]. However, scientific information about the effect of carbon chains geometry as a very basic component of surfactants has not been discussed. However, it was also found that surfactants with a large number of carbon atom masses produced better performance than surfactants with a small number of carbon masses [18]. Unfortunately, the discussion is only about the mass and the number of carbon atoms, while the role of the fatty acid (FA) structure and geometry on the deinking process has not been revealed.

On the other hand, the nature of saturated and unsaturated fatty acid compounds and their structural dynamics are very influential factors in the flotation process. When the mass number of fatty acid grows, the dispersion force between adjacent molecular masses also increases so that the distance of these molecules is closer to each other. [19]. Furthermore, the nature of the hydrophilic-lipophilic balance (HLB) is a parameter that must be considered because surfactants with high HLB values are advantageous during cellulose process and low HLB plays a role in the deinking process [20]. The high or low value of hydrophilic-lipophilic balance is highly dependent on the geometrical structure and the level of saturation of the fatty acid carbon chains. This shows that these factors have the potential to have a direct impact on the deinking process [18]. Therefore, the fatty acid carbon chains contained in

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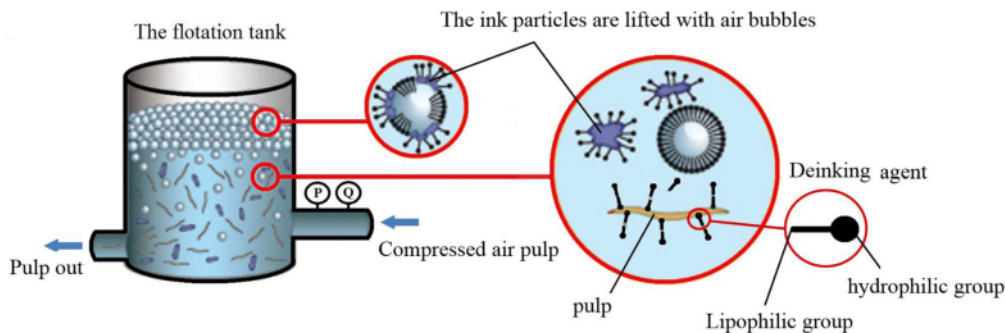


Fig. 1. The research scheme.

**Table 1**  
Fatty acid composition of seed oil of *Morinda citrifolia*.

Affluence(%)	Carbon chains	Weight of molecule	Molecule structure	Cn:db	Paek Quantity (%)
A total ion chromatogram (TIC) = $8 \times 10^4$ of four hours extraction					
15.95	isobutylene	74	$C_4 H_{10} O$	-	8
	butenoic	100	$C_5 H_8 O_2$	1	15
8.57	nitropropane	89	$C_3 H_7 NO_2$	-	Trace
75.27	3 hexenol	100	$C_6 H_{12} O$	1	2
	1,3 dicyclopentyl-2-dodecyl	374	$C_{27} H_{50}$	-	Trace
TIC = $1.5 \times 10^5$ of five hours extraction					
5.54	methyl ethyl cyclohexane	263	$C_{17} H_{28} N O$	-	1
	butyl radoxime	87	$C_4 H_9 N O$	-	Trace
	butanoic acid methyl ester	100	$C_5 H_8 O_2$	1	20
8.16	acetyl hydrazine	74	$C_2 H_5 N_2 O$	-	5
	nitropropane	89	$C_3 H_7 NO_2$	-	Trace
86.3	9, 15 octadecadienoic	294	$C_{19} H_{34} O_2$	2	8
	hexanol	100	$C_6 H_{12} O$	1	1.5
	cyclopropanepentanoic acid	310	$C_{20} H_{38} O_2$	1	0.7
TIC = $9 \times 10^6$ of six hours extraction					
3.26	deutero methyl	298	$C_{19} H_{36} D_2 O_2$	1	Trace
	methyl butanoate	102	$C_5 H_{10} O_2$	-	Trace
96.74	12 octadecadienoic	294	$C_{19} H_{34} O_2$	2	13
	hexanol	100	$C_6 H_{12} O$	1	1
Affluence(%)	Fatty acid	Weight of molecule	Molecule Formula	Cn:db	Peak Quantity (%)
TIC = $4 \times 10^6$ of seven hours extraction					
2	butenoic acid	102	$C_5 H_{10} O_2$	1	1
	isobutyl alcohol	74	$C_4 H_{10} O$	-	25
1.26	pentadecanoic acid	270	$C_{17} H_{34} O_2$	-	15
	tetradecanoic acid	256	$C_{16} H_{32} O_2$	-	25
	methyl heptanoate	144	$C_8 H_{16} O_2$	-	Trace
96.74	12 octadecadienoic	294	$C_{19} H_{34} O_2$	2	18
	14 eicosadienoic	322	$C_{21} H_{38} O_2$	2	0.01
TIC = $14 \times 10^6$ of eight hours extraction					
2.13	pentadecanoic acid	270	$C_{17} H_{34} O_2$	-	15
	tetradecanoic acid	256	$C_{16} H_{32} O_2$	-	25
	methyl heptanoate	144	$C_8 H_{16} O_2$	-	Trace
98.45	12 octadecadienoic	294	$C_{19} H_{34} O_2$	2	20
	14 eicosadienoic	322	$C_{21} H_{38} O_2$	2	Trace

renewable raw materials (noni seed [8]) have the potential to be used as surfactants [19,21]. Unfortunately, scientific information about the role of changes in the carbon chains geometric of fatty acids on the deinking flotation process has not been widely revealed until now [22,23]. The atomic structure of ink particles and triglyceride compounds have the potency to produce potential differences so that increased electronegativity and intermolecular force. These factors allow changing the geometrical structure of triglyceride chains and increase the reactivity of the particles contained in the surfactant so that the ink particles is easy to absorb. Therefore, considering the role of fatty acid carbon chains is very important in the deinking process and there is no scientific information about it, hence the main goal of this study provides detailed scientific information on the role. Moreover, this study used natural surfactants from *Morinda citrifolia* seed oil which were compared with synthetic surfactants. The use of *Morinda citrifolia* seeds is because the seeds often

become waste from processing the fruit which is used as food additives and traditional ingredients. Therefore, this research was conducted to utilize the waste of *Morinda citrifolia* seeds and provide added value to the utilization of the fruit.

## 2. Material and methods

### 2.1. Material

The old newspaper used in this study has a grade 8 which corresponds to the material of previous studies [24,25] and noni seed oil used as a renewable raw material for the natural which was obtained through the Soxhlet extraction process with different extract times, namely for 4, 5, 6, 7, and 8 hours. Furthermore, to determine the diffusion rate of noni seed oil when interacting with ink, Hele-Shaw is used and then

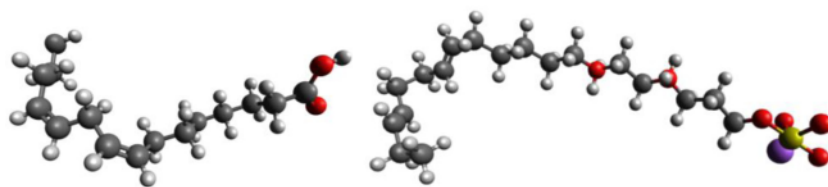


Fig. 2. The carbon chain structure of surfactants. Natural (left); and synthetic (right).

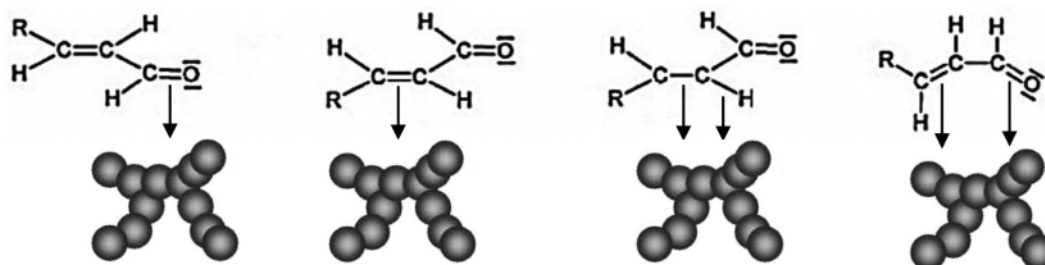


Fig. 3. Molecular attractive force models of surfactants and the agglomerates of black ink molecules.

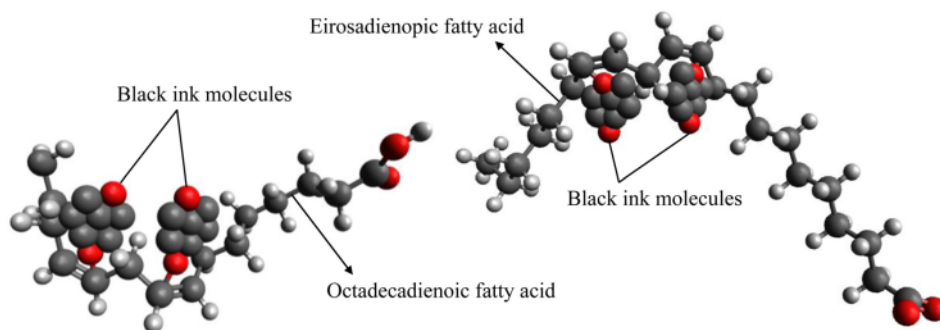


Fig. 4. Molecular interaction of black ink molecules and polyunsaturated fatty acids of natural surfactant (seed oil of *Morinda citrifolia*).

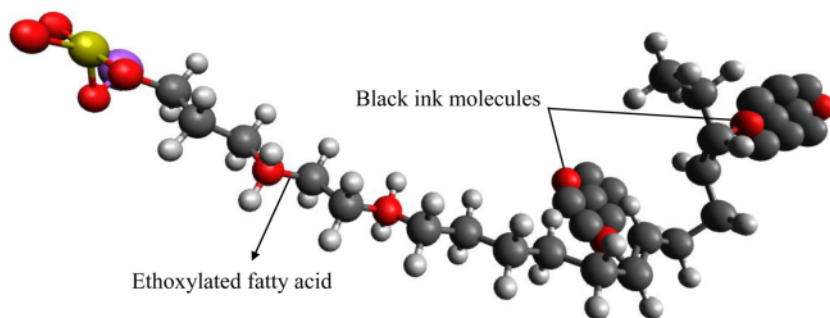


Fig. 5. Molecular interaction of black ink molecules and carbon chain of synthetic surfactant.

compared with other surfactants sourced from palm oil and commercial surfactants.

### 2.2. Method

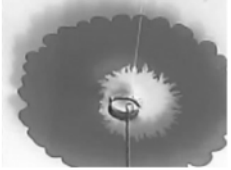

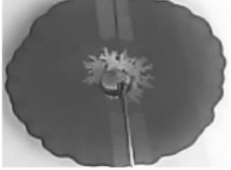


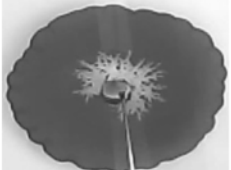




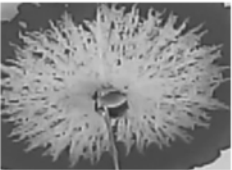
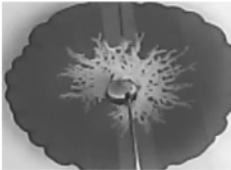
From the experimental scheme (see Fig. 1) it can be seen that in general the test begins by inserting pieces of waste paper into a flotation tank filled with flow water and the air pressure around 1 kg/cm<sup>2</sup>. Furthermore, the noni seed oil is added as a natural surfactant to start the ink removal process in which the ink particles present on the paper are

pulled and adhere to the surface of the water bubbles. The next step is to remove the pulp and form it into a sheet and then measure its brightness and ERIC.

### 2.3. Surfactants roles

Observations were made between two types of surfactants, namely noni seed oil (*Morinda citrifolia oil*) as a natural surfactant and synthetic surfactants to see the response of the molecular structure of the carbon black. A role of the interaction between the ink molecules and the

**Table 2**  
Micro diffusion comparison of various surfactants

Diffusion time	Seed oil of <i>Morinda citrifolia</i>	Synthetic surfactant	Palm oil
24 30 second			
60 second			
90 second			
120 second			

surfactant molecules was observed through the characteristics of deinking performance [17]. It was shown through the resulting brightness and ERIC (see Fig. 6). The composition of fatty acids was determined using gas chromatography mass spectrometry (GC-MS) and the results can be seen in Table 1. In addition, the functional groups possessed by surfactants and the surfactant's ability to absorb energy are known through FTIR testing (seen in Fig. 10). Meanwhile, the phenomenon of attraction between ink particles and surfactant particles was simulated using a chemical computer program. The simulation results can be seen in Figs. 4 and 5.

#### 2.4. Molecular interaction between carbon chain of surfactants and ink molecules

From the GCMS test results in Table 1, it can be seen that the carbon chain structure of natural surfactants is composed and dominated by long chain (polyunsaturated) fatty acid compounds (see Fig. 2). The carbon chain structure of natural surfactants shows an imbalance in the mass of the hydrogen atom in the C=C double bond of the fatty acid carbon chain. This factors has the potential to cause uneven electron pairs resulting in a potential difference between the surfactant and carbon chains of carbon black. This situation has the potential to weaken the dispersion force and increase the molecular interaction of the carbon chain compounds of the ink particles and the surfactant. The weakening of the binding forces between the carbon chains and the presence of unbalanced electron pairs make surfactant molecules have more space to move [26], thus increasing the reactivity of the surfactant molecules, and this has the potential to increase the deinking process so that the paper can be recovered properly. This analysis is followed previous studies which stated that colloidal or hydrophilic colloids have the potential to attract the dispersing medium, which is due to the strong attrac-

tive force between the dispersed particles and the dispersing medium [17,27]. While synthetic surfactants are composed of long carbon chains (see Fig. 2). This makes synthetic surfactants non-polar, more saturated, and stable so that the ink molecules are difficult to dissolve. This phenomenon indicates that nonpolar synthetic surfactants are more saturated than polar natural surfactants, and this conforms with the results of previous studies [28,29] which were discussed from different points of view.

Furthermore, from Fig. 3 we can see that the role of the double bond (C=C) on the surfactant chain triggers intermolecular bonding between fatty acid compounds and ink molecule compounds. Meanwhile, the existence of sulfur trioxide molecules in synthetic surfactant compounds potentially produces attractive forces with water molecules, and the deinking process becomes more effective if it has a low HLB value [20]. The fatty acid of natural surfactants potentially decreased the value of HLB, thus it creates a good penetration characteristic into oil compounds than water, and this is due to the presence of a carboxyl group gives and hydrophilic characteristics of the FAMC compound, and the presence of PUFA in FAMC makes mass carbon atoms increase and function as collector chemicals that are useful in surface reactions between fatty acids carbon chains and ink particles. These factors has the potential to improve the ink removal process and result in higher brightness.

Furthermore, Figs. 4 and 5 shows that there is a molecular interaction between the ink molecules with the FAMC carbon chain and the carbon chain of synthetic surfactant. It can be seen that the oxygen atoms present in the ink molecules can bind to the carbon atoms in the FAMC carbon chain and synthetic surfactants. This is due to the addition of FAMC and synthetic surfactants increase the amount of molecular mass in the flotation column so that the distances between molecules increases and the potential for interaction and attractive forces between ink molecules and FAMC and synthetic surfactant

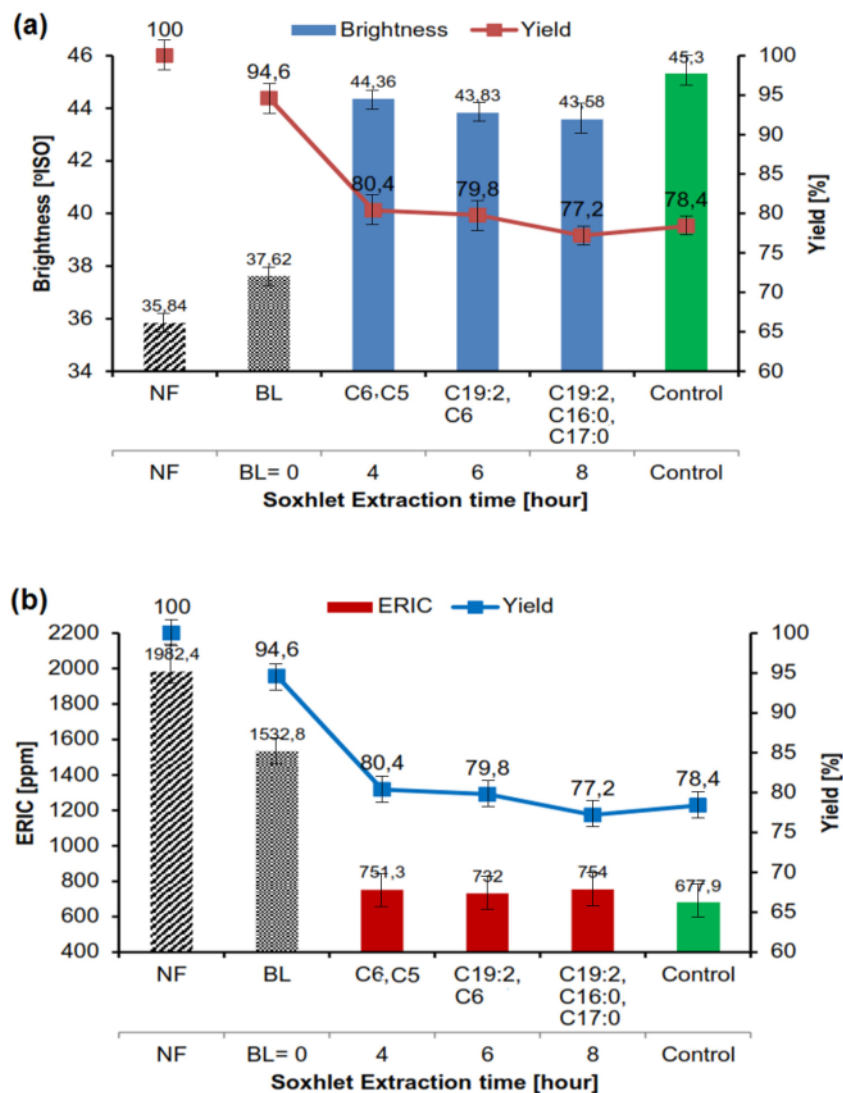


Fig. 6. The effect of deinking flotation with CFAMC. (a). Brightness; (b). ERIC.

molecules is increased. This causes the ink particles to be released from the paper, thereby facilitating the recovery process. The analysis of the effect of molecular density on molecular interactions between carbon chains is consistent with the previous studies [30–32] which also used fatty acid carbon chains and was discussed from another perspective. Moreover, the ability of the fatty acid carbon chains to bind with ink particles is due to the fact that fatty acids function as surface-active chemicals. Therefore they have the ability to act as collector chemicals which are very useful in surface reactions when mechanical force is not effective enough. This analysis is in accordance with previous studies on the effect of carbon chains in alcohol deinking of recycled fibers [21]. This indicates that these fatty acid compounds have the potential as a bond breaker material for ink tissue. If the ink network breaks, it can automatically reduce the mass of the ink network and weaken the bond strength of the carbon chain. This causes the ink to easily slip off the paper and the flotation process is better. Therefore, with the addition of FAMC into the ink particles, and coupled with the presence of a dominant long carbon chain in the FAMC compound (see Fig. 4), the potential

to accelerate the pulping and washing stages thus increases the performance of the deinking process.

### 3. Results and discussion

From Table 2 we can see the effect of natural surfactant on the deinking process. The results show that without natural surfactant the deinking process is not optimal where not all of the ink is released or parted with water. Moreover, it also appears that the water is not too bright. However, with natural surfactants, it can be seen that more ink is released, which is indicated by the large area of water brightness. This phenomenon shows that natural surfactants play a powerful role in breaking bonds in ink networks, which can help to detach print from paper fibers. These results indicate that the interrelation of fatty acid with ink molecules is truly affected by the presence of hydrophobic molecules and frothing agents. This analysis is following the results of previous studies [33]. These results indicate that the long carbon chain and the short and unsaturated carbon chain must take a role like soap molecules

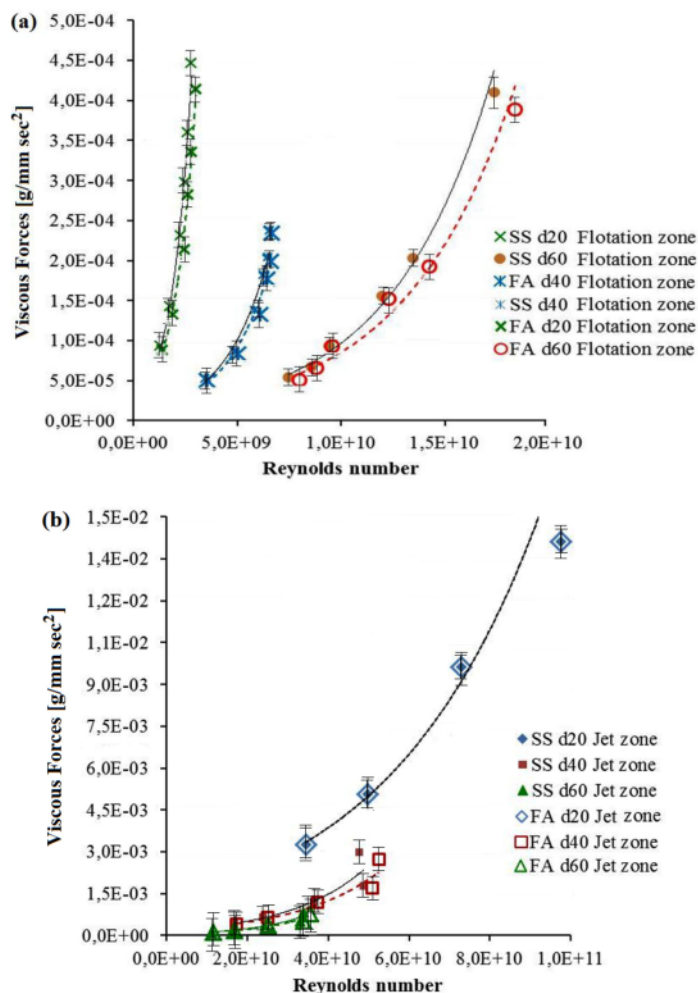


Fig. 7. Viscous forces of bubbles in flotation (a) and jet region (b).

with their polar compo<sup>27</sup>. Moreover, this phenomenon also shows that the effect of increasing the number of carbon atoms and the bent geometry of the carbon chain due to the presence of the double bond and a short carbon chain with a small molecular mass. <sup>11</sup> has the potential to increase atoms reactivity so that hydrogen bonding with oxygen and the hydrogen atom of ink particles potentially occurs.

Furthermore, the composition of *Morinda citrifolia* fruit seeds consists of several <sup>12</sup>g and short-chain fatty acid compounds, namely polyunsaturated fatty acids, polysaturated fat<sup>23</sup> acids, unsaturated fatty acids, and saturated fatty acids (see Table 1). It can be seen that the fatty acid composition of *Morinda citrifolia* fruit seeds is dominated by polyunsaturated fatty acids (C19:2), which is around 90-95%. The <sup>6</sup> long and bent C19:2 structure is due to it has 2 double bonds that have the potential to produce a higher brightness. This analysis is possible because the molecular mass is a function of the attractive forces of molecules, thus when the FAMC carbon chain is dominated by C19:2, the molecular mass automatically increases. This causes the FAMC molecules to be more positive and energetic than the ink particles which have a lower molecular mass. Therefore, with the potential difference between the two compounds, the ink particles are easily attracted by the FAMC carbon chain so that the ink release process becomes more effective. This is evident from Fig. 6a, where it can be seen that FAMC has a fairly good brightness

value, which is around 43 to 44. Moreover, Fig. 6b also shows the Effective Residual Ink Concentration (ERIC) of deinked <sup>28</sup> after flotation with FAMC. On the other hand, it is also seen that the performance of synthetic surfactants in the non-flotation deinking process (NF) and the flotation process (BL) is lower than natural surfactants. Moreover, from the observations, it can be seen that the FAMC C5 and C6, and C19:2 and C6 carbon chains produced the best brightness and ERIC, and when compared with the performance of synthetic surfactants, the FAMC C5 and C6, and C19:2 and C6 carbon chains had almost similar performance. This is due to FAMC and synthetic surfactants are composed of the same fatty acid compound, namely a long and bent polyunsaturated carbon chain. The imbalance of molecular mass and the existing sigma and pi orbitals in the double bond potentially increase the reactivity of atoms, which the electron reactivity is potentially greater in the phi orbitals. This is very possible because the electrons in the phi orbitals have a larger space to move. After all, the distance between the phi orbitals and the atomic nucleus is greater than the distance between the electrons in the sigma orbitals and the atomic nucleus.

However, from Fig. 8 it can be seen that the best brightness and ERIC values were produced by short carbon chains (C5 and C6) and not by C19:2, C16, and C17 groups. This phenomenon shows that the brightness is not only achieved when the number and length of carbon

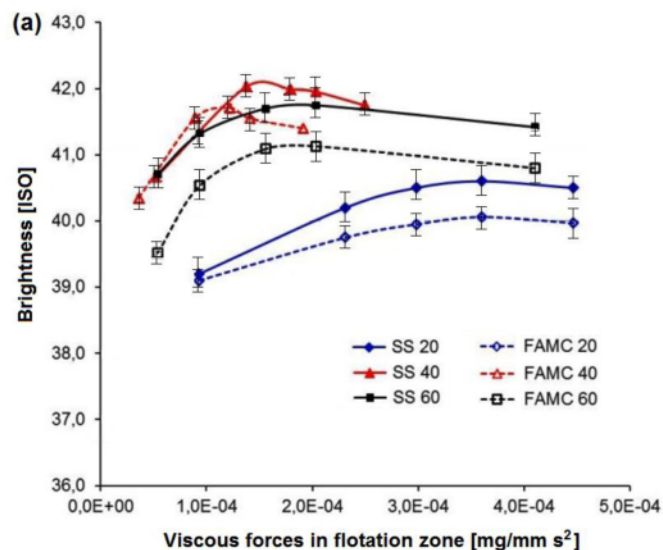
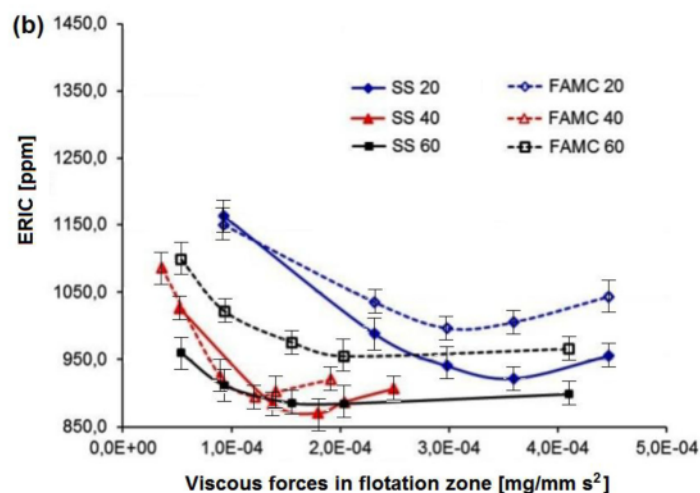


Fig. 8. The viscous forces effect of brightness (a), and ERIC (b).



atoms increases but can also be achieved by short carbon chains with a small mass of carbon atoms. This result is very different from previous studies which said that the brightness can be achieved if there is an increase in the mass number of the carbon atoms in fatty acids [21]. This analysis is very possible because although the C5 and C6 carbon chains are included in the saturated fatty acid group, the small number of atomic masses makes them unsaturated and polar. The phenomena are conformable to our prior research using three straight vegetable oils (coconut, jatropha, and sunflower) which were discussed from different perspectives [28,34]. This is very possible because the small mass of atom carbon in fatty acids potentially weakens intermolecular binding forces. This factor makes C5 and C6 more reactive and can move randomly and increases the chance of an effective collision so that the potential for interactions and bonds between the fatty acid carbon chains and the ink molecules is increasing. This has the potential to improve the deinking process so that the ink release process becomes better.

Moreover, from the observations, it was also found that the length of hydrocarbon chain (unsaturated and saturated structures) will create the way of interaction during deinking flotation. The length of chain of hydrocarbon (polyunsaturated) is the crucial actor to compose hydrophobicity nature for surfactant.

From Fig. 7, it can be seen that the compressed air trapped in the bubble produces a viscous force. This causes the released ink to be lifted in the flotation area, while in the jet area there is a release of ink from the fiber surface. These results indicate that viscous forces are important in this analysis as per the results of previous studies [8,35]. The explanation of this phenomenon is described more clearly in Fig. 8.

From Fig. 8 it is clear that the brightness and ERIC due to deinking flotation are strongly influenced by the viscous force. It can be seen that the bubbles produced by synthetic surfactants occur at a maximum viscous force of about  $1.5\text{--}2.0 \times 10^{-4} \text{ mg/mm}\cdot\text{s}^2$ , on the other hand, the tail of the carbon chain methyl ester a viscous force around  $1.0\text{--}1.5 \times 10^{-4} \text{ mg/mm}\cdot\text{s}^2$ . These results indicate that the viscous force



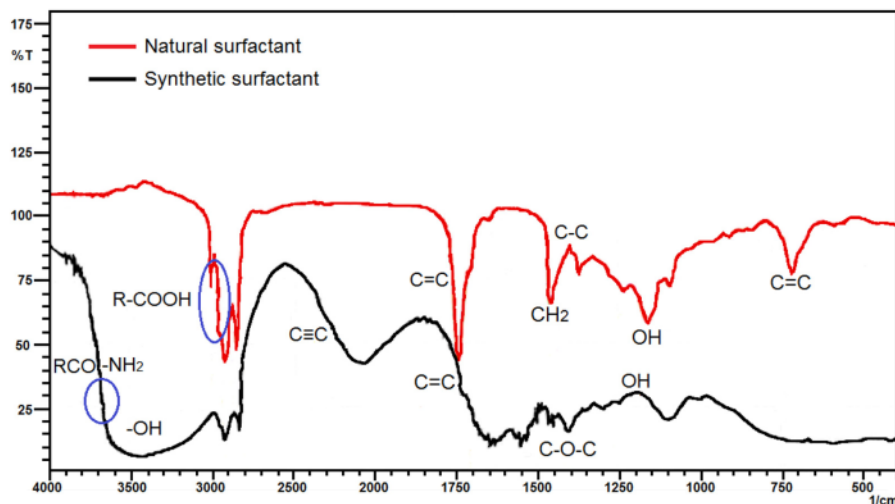


Fig. 9. Energy absorption of natural and synthetic surfactant.

that occurs in synthetic surfactants is greater than the viscous force that occurs in FAMC. This phenomenon proves that the intermolecular binding force of the synthetic surfactant is stiffer than the intermolecular bonding force in the FAMC system. However, an important factor that must be considered is that the resulting viscous strength should not exceed or be too large. This should be considered to prevent the surfactant's intermolecular bonds (or FAMC) with the ink particles potentially breaking, leading to suboptimal flotation removal results, lower brightness, and higher ERIC.

From the results of the previous discussion based on the resulting study in Figs. 7 and 8, it is known that the natural surfactant FAMC has a lower viscous force than the synthetic surfactant. Based on the previous results, the role of natural synthetic FAMC and synthetic surfactants in the deinking process can be explained as follows: The bond enthalpy contained in each molecule is fixed, however, if they get energy, the atomic volume potentially increases so that the distance between the electron and the atomic nucleus increases which has the potential to weaken the van der Waals force between atoms. When this happens, the energy needed to break molecules becomes less so that the process of absorption of energy by molecules improved. The result shows that at the similar molecules, the tip of FAME is higher than synthetic surfactant, and the heat energy of IR is absorbed by FAME around 109 %T, while that of synthetic surfactant is around 88 %T.

These results prove that natural FAMC surfactants have great potential to weaken the dispersion forces that bind the ink particles thereby increasing the reactivity of the molecules. Moreover, this proves that natural FAMC surfactants have better performance than synthetic surfactants. This analysis was confirmed by the better brightness of natural surfactants than synthetic surfactants (Fig. 6), and the lower viscosity of FAMC natural surfactants than synthetic surfactants (Fig. 8). Moreover, the differences in behavior and response of natural surfactant molecules and synthetic surfactants to energy are not on the saturation of the carbon chain, however, due to the amount of atomic mass (see Table 1) and the structure of the hydrocarbon chain. (see Figs. 4 and 5). Moreover, these results indicate that surfactant performance is not only influenced by colloidal forces [27], bubble size, surface tension, and the amount of surfactant dose [8,22,36], but there is another fundamental scientific thing, namely the ability of surfactant molecules to absorb energy which is a function of the geometric of the hydrocarbon chain and the amount of molecular mass.

#### 4. Conclusions

The role of *Morinda citrifolia* oil for deinking process to recovered papers in a flotation column was performed, and the prominent findings were as follows:

- 1 The carbon chain structure of natural surfactants and synthetic surfactants has the potential to cause uneven electron pairs resulting in a potential difference between the surfactants and carbon chains of ink particles. This situation potentially reduces attractive interactions between molecules and weakens the van der Waals forces thereby increasing the reactivity of the surfactant molecules and increasing the deinking process so that the paper can be recovered properly.
- 2 The ability of fatty acid carbon chains as surface-active chemicals increases the strong intermolecular interaction with the ink particles so that the ink easily slips off the paper and the flotation process is better. Moreover, the presence of FAMC into the ink particles, and coupled with the presence of a dominant long carbon chain in the FAMC compound accelerates the pulping and washing process thus increasing the performance of the deinking process.
- 3 The synthetic surfactant compound was more saturated than the natural surfactant. Therefore the natural surfactant FAMC has a lower viscous force than the synthetic surfactant. This result proves that the natural surfactant FAMC can weaken the bond of ink molecules and increase the reactivity of molecules. Natural surfactants have better performance than synthetic surfactants.
- 4 During the deinking process, it was seen that the natural surfactants had a better brightness than synthetic surfactants. Synthetic surfactant compounds are stiff so that the ink particles are difficult to separate. This phenomenon indicates that the intermolecular binding forces of synthetic surfactants are stronger than natural surfactants. These results indicate that the deinking performance of natural surfactants is better than synthetic surfactants Fig. 9.

#### 5 Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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