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The role of functional group of fatty acids in *Morinda citrifolia* L. on surface tension and diffusion performance into ink particles

Abstract

The interaction of fatty acid of *Morinda citrifolia* L. (FAMC) with ink was studied using Hele Shaw Cells. The interaction is compared to that of fatty acid derivate of synthetic surfactant. These interactions are modeled and explained through micro diffusion and its viscous fingering character. The results show that the structure of the molecule and the number of double bonds has a special effect on surface tension and their micro diffusion character. FAMC at six-hour extraction has the highest speed of interaction and highest perimeter of diffusion among synthetic surfactants and other fatty acids in *Morinda citrifolia* L.

Keyword: molecular structure; fatty acids; surfactant; *Morinda citrifolia* L.; ink particles; diffusion

1. Introduction

The use of surfactant in deinking flotation has been studied by many researchers [1-4]. Surfactants for deinking flotation are primarily anionic and non-ionic surfactants. They can be used effectively for deinking flotation because of their ability to interact with water (has hydrophilic properties) and oil (has lipophilic properties) [2]. Most research of deinking flotation are mainly concern about the effect of ionic properties, HLB value, or degree of polymerization of surfactants / synthetic surfactants used in the deinking flotation [1-2], however, the interaction between surfactant and ink has hardly ever been discussed. The performance improvement of deinking flotation requires better understanding on this interaction.

Fatty acid based surfactants contain certain poly un-saturated fatty acids (PUFA) such as C16:0, C18:0, C16:1, C18:1, C18:2, C18:3 [5]. According to Khalek [1], the longer the hydrocarbon chain, and the greater the number of un-saturated structures, the better the surfactant performance will be for the deinking flotation process. Some surfactants are fatty acid derivate. Its hydrophilic properties depend on how its carboxylic part has been modified to have ionic properties. Its lipophilic properties depend on the structure of the hydrocarbon chain [5-7]. A fatty acid is immiscible with water. The shorter the hydrocarbon chains of a fatty acid, the better its solubility in water; in other case, the longer the hydrocarbon chain, the less their polarity. The hydrocarbon structure of a fatty acid, and how it has been modified, determines the polarity and accessibility into the ink particle. This suggests that the different polarity of a hydrocarbon chain will promote different action in micro diffusion. The greater the electronegativity differences between atoms in a bond, the more polar the bond. The combination of carbons and hydrogen, as in hydrocarbons or in the hydrocarbon portion of a molecule with a functional group, is always non-polar. The polarity of the functional group are very different, depend on the modification that has been completed [8].

Micro diffusion in Hele Shaw cells has been used to analyze the molecular displacement between two fluids with different viscosity [9-14]. The influence of viscous fingering phenomena, and how it increases various mobility ratios and injection speeds, is explored [15]. How the fluid–to–fluid is displaced between the more and less viscous of fluids, consisting of fully miscible fluid, immiscible fluid, and different Newtonian fluid properties has been observed [16].

Studying the micro diffusion involving chemical reactions between two miscible fluids shows that at different patterns, the density area of the pattern and the times needed to achieve the same radius and maximum radius are also different [12]. Other research involving viscous fingering exhibited the same mobility ratio and shows that different concentrations produced different patterns [11]. This proves that the diffusion process is a function of viscosity gradient and concentration gradient. Substances that have difference lipophilic properties will shows difference character in their interaction with other substances. In deinking flotation, the use of surfactants with difference properties (difference lipophilic properties or hydrophilic properties), or in other word has difference HLB value should shows difference character in their interaction with inks. The research work on this phenomenon in the deinking flotation is hardly found, so it is elucidated in this study.

This study reveals that synthetic surfactant and the fatty acid of *Morinda citrifolia* L. (FAMC) have different polarity and lipophilic properties that exhibit different patterns and qualities of viscous fingering. How the surfactant works in deinking flotation is elucidated and modeled. The interactions were studied using Hele Shaw cells to identify the diffusion interaction between the surfactant and ink particles on the micro scale. The interactions between synthetic surfactant and FAMC were also studied.

2. Materials and methods

2.1 Materials

The materials used for the diffusion experiment include FAMC, synthetic surfactant (fatty acid derivate) from KAO Industrial estate supplied by PT Lautan Luas Indonesia, cadmium red – Oil base ink (Graphic Chemical and Ink Company), n-hexane (technical grade – 95% purity from Smark Lab), vehicle oil (Castor oil from Palma Christi) and mineral spirits (paint thinner– from Merck) procured from local vendor.

2.1.1 FAMC preparation

The FAMC was extracted from the seed of *Morinda citrifolia* L. using n-hexane. The seed was collected and separated from ripe plants that are abundant in the East Java Province in Indonesia. The seed was dried and then made into powder. The extraction processes were performed for either four, five or six hours in a Soxhlet extractor. The extracts of FAMC were concentrated (to evaporate the n-hexane) in rotary vacuum evaporator of IKA@ RV 10 digital.

2.1.2 Dilute ink preparation

Vehicle oil and mineral spirits (paint thinner) were used to dilute the paste ink with ratio of 30 % volume of paste ink, 30 % volume of vehicle oil, and 40 % volume of mineral spirit to facilitate injection into the Hele Shaw cell equipment as shown in figure 1.

2.2 Methods

2.2.1 Diffusion Experiment

The Hele Shaw cell was constructed by creating a narrow gap with a flat glass of a plate with a small hole of 5 mm in diameter. The red ink was injected into the cell until 3.0 cm diameter of spreading ink was achieved. The next step, the surfactant was injected (drop by drop) into the hole of the Hele Shaw cell through a syringe as shown in Figure 1. The injection pressure of the syringe pump was produced using an adjustable weight. The adjustable weight was kept at 480 gram for all experiments. The surfactant injected into the ink performed viscous fingering due to the gradient of surface tension, viscosity, and concentration. The hydrodynamic nature of the viscous fingering was measured and recorded using a camera with a macro lens and then was analyzed with image J to observe its characteristics. The molecular structure of FAMC and synthetic surfactant was clarified.

(Figure 1)

2.2.2 Diffusion analysis

In the interaction between two phases of fluids, diffusion always happens. Consequently, when the two phases of fluids are connected, such as when the surfactant and ink particles are used in deinking flotation, they are not in equilibrium. The system attempts to reach equilibrium by a relatively slow diffusive movement of the constituent parts, which transfer

in part between the phases in the process. The fluid substances are moving from a place of high concentration or of high viscosity to one of low. The rate at which a substance moves at every point in any direction depends on the gradient of potential energy (viscosity gradient or concentration gradient).

$$J_A = -D_A \frac{d\,c_A}{d_x} = -C_A M_A \frac{d\mu_A}{d_x} \rightarrow J_A \approx L_A \frac{d\mu_A}{d_x} \tag{1}$$

In this case:

$$D_A = C_A M_A \frac{d\mu_A}{dc_A} \approx -L_A \frac{d\mu_A}{dc_A} \tag{2}$$

$$L_A = -C_A M_A \tag{3}$$

In this case (with respect to other molecule):

$L_A = Potential\ energy\ gradient\ of\ molecule\ A$

$M_A = Mobility\ of\ molecule\ A$

$C_A = Concentration\ of\ molecule\ A$

$D_A = Diffusivity\ of\ molecule\ A$

$\mu_A = Viscosity\ of\ molecule\ A$

$J_A = Flux\ of\ molecule\ A\ relative\ to\ the\ average\ molecular\ velocity$

It can be seen from equation (1) through (3) that the micro diffusion of molecule A, relative to other molecules in the system, is dependent on the energy potential gradient. The characteristics of micro diffusion were analyzed and evaluated by measuring the speed of interaction between the ink and FAMC, and between ink and synthetic surfactant.

$$Speed\ of\ interaction = \frac{millimeter\ of\ perimeter\ of\ micro\ diffusion}{times\ needed\ for\ conducting\ micro\ diffusion} \tag{4}$$

Whereas the perimeter was defined as: the measured length of the interface between ink area and FAMC or synthetic surfactant during micro diffusion.

3. Results and discussion

3.1. FTIR of FAMC and synthetic surfactant

From figure 2 it can be suggested that the structure of FAMC is fatty acid with alkane and alkene structures, and its functional group is carboxylic acid. In case of synthetic surfactant, it

has also alkane, alkene and alkyne structure. In its functional group, it has amide and alcohol structure. If we see on the infrared absorption peak between 3200 – 3700 it seem has a smooth tongue shape in the lower wavenumber zone (3200 – 3500 cm^{-1})but it also has several vampire fangs (with a few higher percentage of transmittance) in the higher wavenumber zone (3500 – 3700 cm^{-1}). So it can be assumed that the carboxylate functional group of tatty acid origin (usually in the range of 2700 – 3000 cm^{-1} wavenumber) of synthetic surfactant has been modified into amide structure that contains alcoholic structure as its functional group R-NH-CH$_2$OH.

3.2. Effect of molecular structure of FAMC and synthetic surfactant on the interaction with water molecules.

The molecular structure of synthetic surfactant, FAMC, and ink molecules that contains red-cadmium dyes and drying oil are illustrated. However, the intermolecular bonding between water molecules and synthetic surfactant and between water molecules and FAMC were derived on the hydrogen bonding principles and polarity of any part of synthetic surfactant and fatty acid molecules.

(Figure **3**)

Based on this theory, it can be seen that synthetic surfactant has better solubility than FAMC as illustrated in figures **3** and **4**. According to the molecular interaction model, synthetic surfactant has good accessibility into water molecule but FAMC does not. Synthetic surfactants have possibility creating hydrogen bonding with more than 4 water molecules up to (4 + 4n) water molecules whereas n is the numbers of ethylene oxide presence in their structure. In this case FAMC has possibility to create 5 hydrogen bonding. When it comes into contact with water, the ionic group being highly polar and strongly attracted to water molecule whereas the hydrocarbon part that has lipophilic properties break – up the local hydrogen bonds and creating lower surface tension.This theory has supported other research in the use of surfactant in fire extinguisher such as ethoxylated fluorosurfactants and perfluorinated sulfamats that able to reduce aqueous solution surface tension to lower value of 15 – 20 mN/m. Both of these surfactants are surface active in aqueous solution and has considerably higher surface active than hydrocarbon surfactants at similar critical micelle concentration (CMC). Replacement of only one fluorine with hydrogen in the terminal CF3 group results in a substantial decrease of surfactant effectiveness [18]. The fluorine as well as oxygen in [EO]$_n$ has free electron pair which made them to have strong tendency to have hydrogen bond with water molecules for better hydrophilic character. This evidence has also been discussed in other research. It is reported that by changing the hydrophilic part (by adding spacer of one, two or three carbon atom in their hydrophilic part) of surfactants that have the same length of hydrocarbon tail affect the flotation recovery of calcite – fluorite minerals [19]

(Figure**4**)

3.3. Effect of molecular structure of FAMC and synthetic surfactant on the interaction with the ink molecules.

Both synthetic surfactant and FAMC shows good infiltration into the ink particles.The ink particles are composed of cadmium red dyes and drying oil (fatty ester). The vehicle and solvent are believed to evaporate. These show that the combined structure of cadmium red dyes and drying oil are responsible for the interaction of ink particles with synthetic surfactant and FAMC. In the case of an interaction between the ink particles and synthetic surfactant or FAMC was established, the lipophilic structure of synthetic surfactant and FAMC interacts with the ester part of drying oil (ink particles). The dried ester portion of the ink particles are dissolved in the synthetic surfactant or FAMC according to the "like–dissolved–like" principle as it is discussed elsewhere.

In this research, the molecular interaction takes place in the oil phase only. The model of accessibility between the FAMC and synthetic surfactant into an ink particle was compared. FAMC is composed of short chains of hydrocarbon (product of four-hours of Soxhlet extraction) and long chains of hydrocarbon (product of six-hours of Soxhlet extraction). FAMC with short chain of hydrocarbon has lower lipophilic character than FAMC with long chain of hydrocarbon. FAMC at four-hours just has possibility to interact with one molecule of an ink particle, whereas FAMC at six-hours has the possibility tointeract with two molecules of ink particles. This all has correlation with its HLB value [17].

As presented in figure **3**, each ink particle has the possibility to have interactions with eightmolecules of FAMC with the long chain of hydrocarbon that has two double bounds, and it has possibility to have interactions with four molecules of FAMC with short chain of hydrocarbon that has only one double bounds. As seen in figure **4**, each ink particle has the possibility to have interactions with eight molecules of synthetic surfactant with long chain of hydrocarbon that has two double bounds. This indicates that the molecular structure of the lipophilic part of FAMC is the same (or at least similar) with synthetic surfactant, that is, the accessibility of both of them into ink particle is almost the same (similar).When considering the interaction between the lipophilic part (hydrophobic part) of FAMC or synthetic surfactants with the hydrocarbon substances (ink), the interaction characteristic can be considered such as in the case of the spreading of hydrocarbon surfactant or fluorosurfactant on lipophilic substances (such as ink). In this case the speed of interaction of FAMC of 6 hours is approaching of that of synthetic surfactant, and far higher than that of FAMC of 4 hours.The lipophilic properties (speed of interaction) of FAMC of 4 hours (that contain short hydrocarbon chain of fatty acids) are lower than that of FAMC of 6 hours and synthetic surfactant. This is supported by the fact that the speed of spreading of fluorosurfactant (that contain oil repellant) over a hydrocarbon substance is far lower than that of hydrocarbon surfactant. Spreading in this case is determined by a transfer of surfactant molecules on a bare hydrophobic substrate in front of the moving three-phase contact line (autophilic phenomenon) [18].

3.4 Effect of molecular structure of surfactants on its micro diffusion character.

Micro diffusion between two fluids that has differences in their energy potential gradient has a close relation with its viscous fingering character. The speed of fingering and the length of the perimeter of a fluid that is displaced by another fluid that has energy potential gradient were used to interpret the interaction between synthetic surfactant – and –ink particle, and between FAMC – and – ink particle.

(Figure 5)

As shown in figure 5, FAMC products of Soxhlet extraction of four, five and six hours were evaluated for their micro diffusion characters and the results were compared with the micro diffusion characters of synthetic surfactant.The FAMC product at four-hours showed the worst fingering pattern as compared with other FAMC. The FAMC product at six-hours showed the strongest full micro diffusion character (fingering) to push, penetrate, and displace the ink area. The synthetic surfactant is able to diffuse directly (good penetration without high effort to push) and displace a few inkareas. This phenomenon seem correlate with its behaviors to construct intermolecular bonding (Van der Waals) between hydrocarbon that interfered by its molecular structures [20]. All of the viscous fingering patterns are dendritic. These dendritic patterns show thatthe FAMC product at the six-hour extraction is the most suitable surfactant for ensuring good penetration into the ink structure. The synthetic surfactant has better infiltration into the ink area, and it has a small area of displacement around the injection hole.This is suggested because of the presence of alkyne structure in synthetic surfactant (see at FTIR analysis in figure 2). When this synthetic surfactant is comes into contact with ink molecule micro diffusion start immediately without any lead time.

3.5 Micro diffusion characters of FAMC and synthetic surfactant.

To understand the differences of micro diffusion character among them, the speed of interactions and its perimeter was evaluated.

(Figure 6)

From figure 6 it can be inferred that FAMC at six-hours provides the highest speed of interaction than the others. This means that the interaction capability of FAMC product at thesix-hour extraction point is the best and far better than that of the synthetic surfactant.

(Figure7)

In figure 7, it is clearly seen that the perimeter of diffusion of FAMC at six-hours is still higher than that of the synthetic surfactant. The perimeter of diffusion and the speed of interaction of synthetic surfactant is more or less the same as FAMC at five hours of Soxhlet extraction. From this condition, it can be suggested that the lipophilic part of synthetic surfactant is composed of a fatty acid that is similar in character with the fatty acid presence

in the FAMC at five hours Soxhlet extraction, but it doesn't mean that the molecular structure is quite the same.

(Figure **8**)

The area occupied by the fingering pattern of synthetic surfactant around the injection hole is less than that occupied by FAMC (Figure **5**). However, from figure **8**, it is shown that synthetic surfactant has much higher ratio of perimeter to area. This shows that synthetic surfactant is able to infiltrate directly into the ink area, but FAMC does not. The finger width of the pattern of synthetic surfactant is very narrow. This means its accessibility into the ink structure is better than FAMC due to its better accessibility to water as illustrated by the model in figures **3** and **4**. Based on our best knowledge, these all may have a close relationship to their polarity. As it was discussed elsewhere [8]: the polarity of a fatty acid (FAMC) and the fatty acid derivate depends upon the length of the hydrocarbon, the number of doublebonds, the presence and number of modified parts of the hydrocarbon chain and the functionally group [8, 1].

Every fluid has its surface tension. When fluids come into phase contact with each other, their surface tension will change. So in viscous fingering, it should be a surface tension changes in line with its micro diffusion. In their interaction, there are inconsistencies in their speed of interaction, perimeter of diffusion and perimeter/area of diffusion as a function if times (Figures 6, 7 and 8). In attempt to reach equilibrium by transferring in part between the phases the rate at every point and direction depend on the potential energy (such as viscosity gradient, concentration gradient, and also surface tension gradient). This fact was supported by Taylor Saffman theory that surface tension should be included in their Hele Shaw Cells equation [21].The finger width of viscous finger for synthetic surfactant is narrower than that of FAMC, this mean the finger width depend on the surface tension because the surface tension between synthetic surfactant and FAMC and even between FAMC itself is difference even though the surface tension of them did not measured. This finding agree with other research finding, that is, the effect of surface tension on finger width is not quantitatively accurate, but qualitatively its behavior is correct [22].

4. Conclusions

Extraction times have significant effect on the molecular structure of FAMC constituent. The six hours of soxhlet extraction time gave the most suitable FAMC product for deinking flotation surfactant. The interaction capability of FAMC product at the six-hour of extraction is the best and far better than that of the synthetic surfactant and other FAMC product. This can be inferred by its micro diffusion characteristic, such as the speed of interaction and its perimeter of diffusion.

The speed of interaction and the perimeter of diffusion of FAMC at six hours of soxhlet extraction time are the best as it is compared to the others. It has the highest speed of interaction than that of synthetic surfactant and other FAMC products. It also has the highest

perimeter of diffusion, far higher than what is exhibited by other FAMC products and synthetic surfactant, but its accessibility into the ink structure is lower than that of synthetic surfactant.

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The role of the molecular structure of fatty acids in *Morinda citrifolia* L. and syntheticsurfactant to increase good diffusion into ink particles

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e-mail address: trismawati@upm.ac.id

W: adjustable weight
C: camera (macro lens)
S: syringe
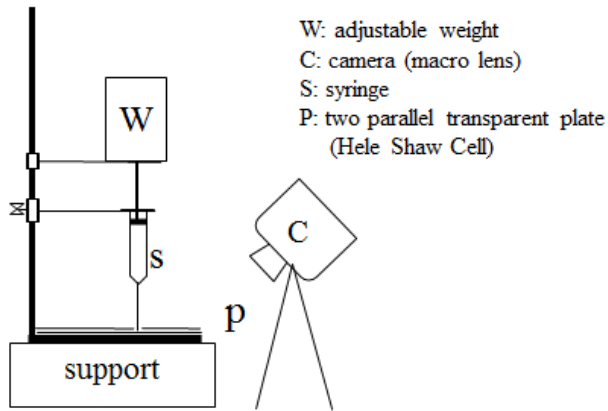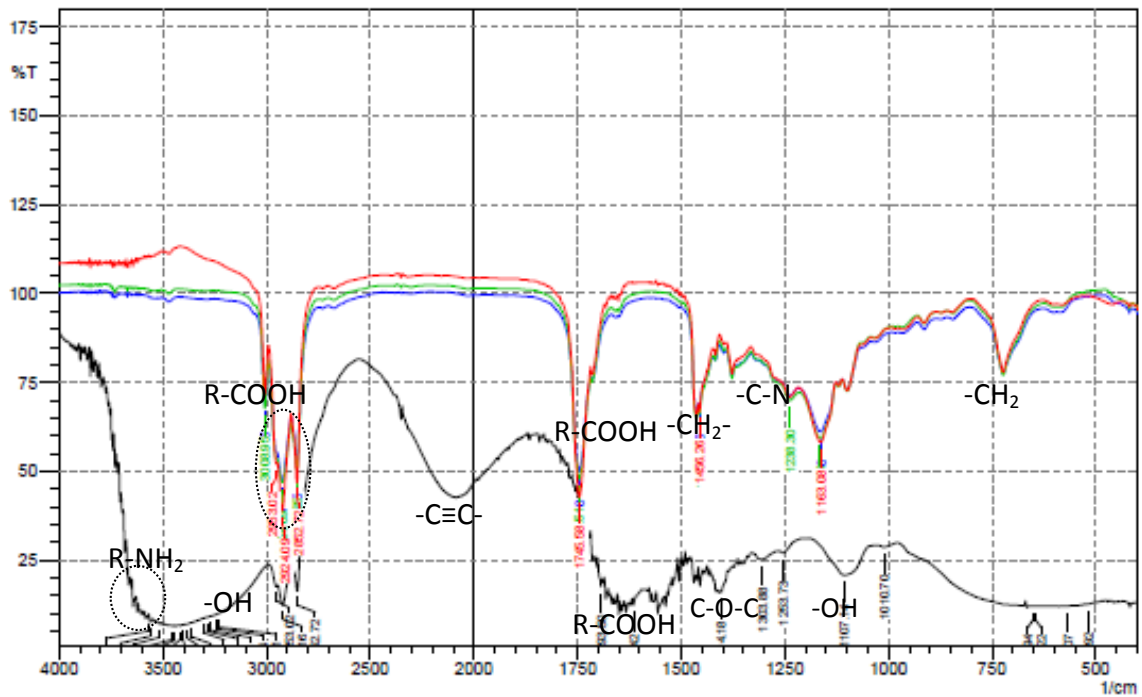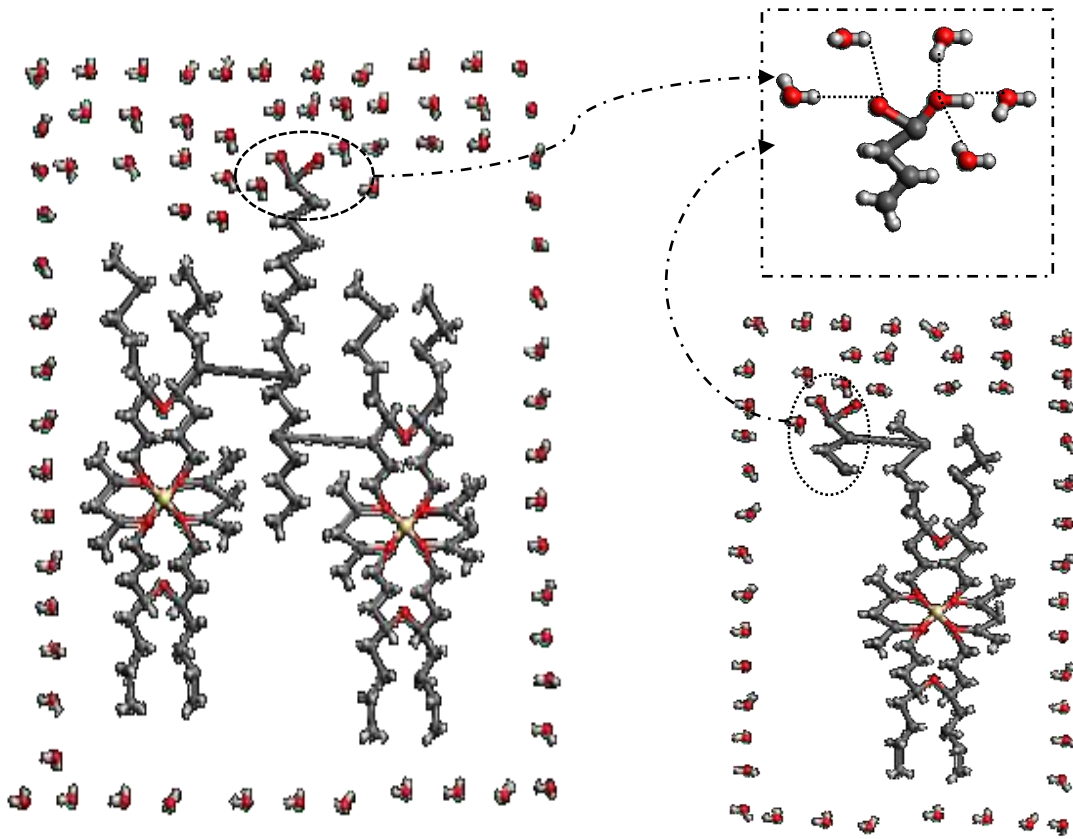P: two parallel transparent plate
    (Hele Shaw Cell)

Figure 1. Experimental Equipment

Figure 2. FTIR Analysis of FAMC and synthetic surfactant

FAMC of 6 hour soxhlet extraction:

91 % [C18:2 fatty acid structure] + 9 % [alcohol structure, H3C...OH]

FAMC 4 hour soxhlet extraction:

47 % [short chain fatty acid structure] + 29 % [alcohol structure, H3C...OH]
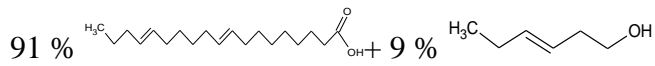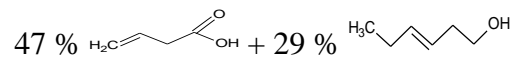
Figure 3. The interaction between (a) ink molecules of cadmium red ink with C18:2 of FAMC as the long chain of fatty acid, and (b) ink molecule of cadmium red ink with C_5H_9-COOH of FAMC as the short chain of fatty acid in the surrounding of water molecule. Insert: interaction of the functional group of fatty acid with water molecule.
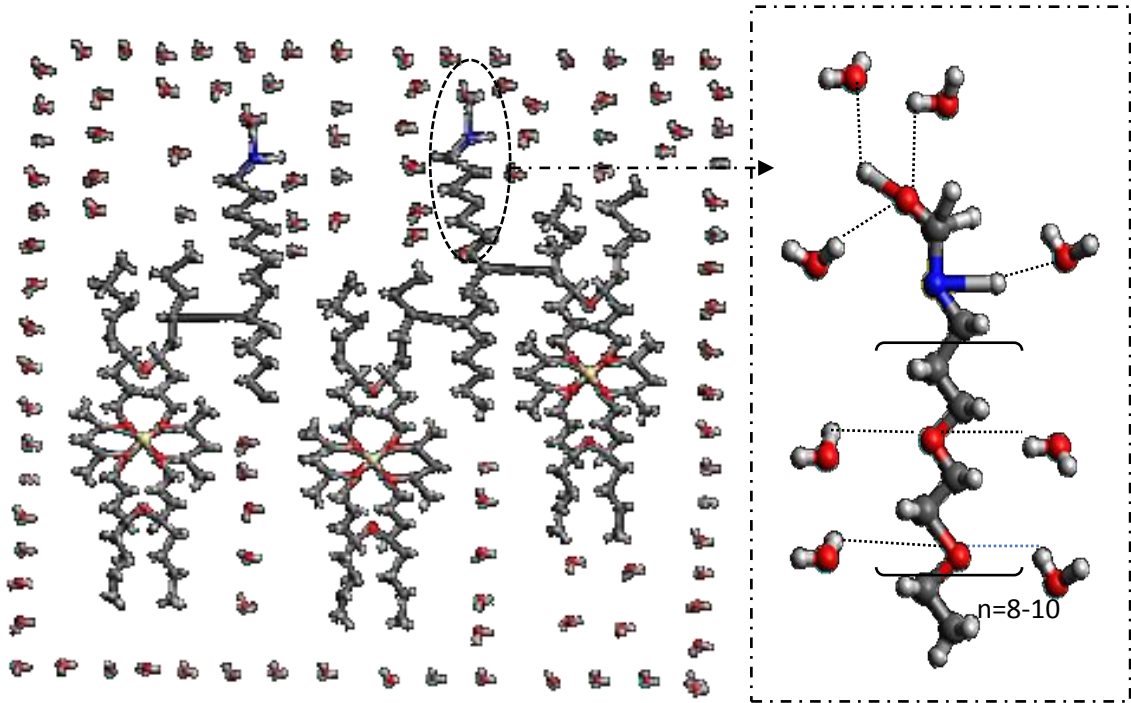
Synthetic Surfactant (Derivate of Fatty acid): mostly a mixture of C16:1 (left) and C18:2 (right).

Functional group of synthetic surfactant

Figure 4. The interaction between ink molecules with synthetic surfactant (that consist of ethoxylated C16:1 and C18:2) in the surrounding of water molecule. It is also shown the interaction between red cadmium inks with synthetic surfactant. Insert: interaction between the functional group of synthetic surfactant with water molecules
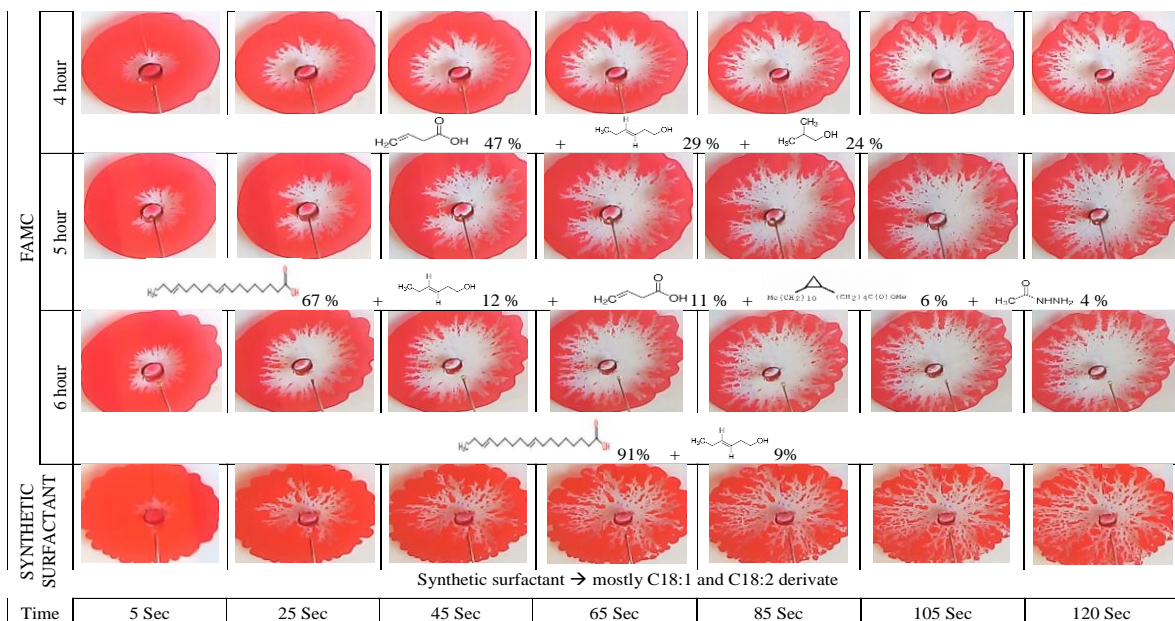

Fig. 5. The pattern of viscous fingering of FAMC and synthetic surfactants in ink particles
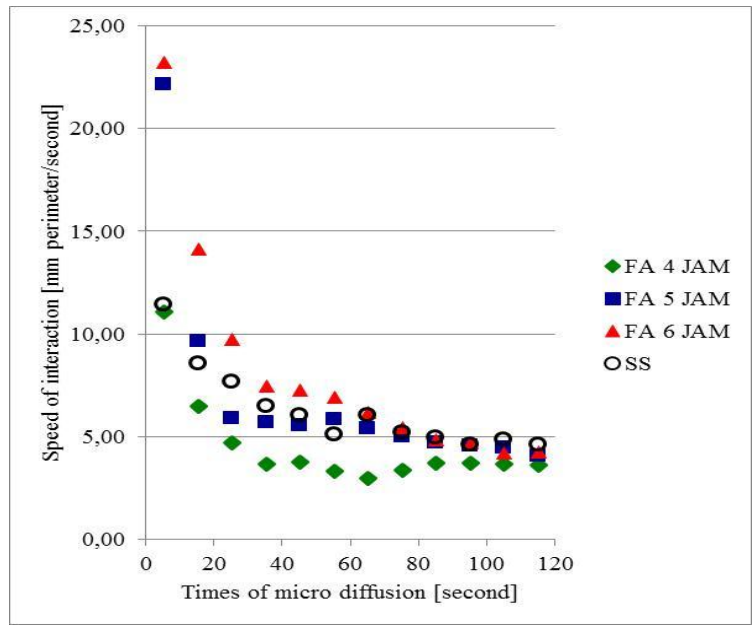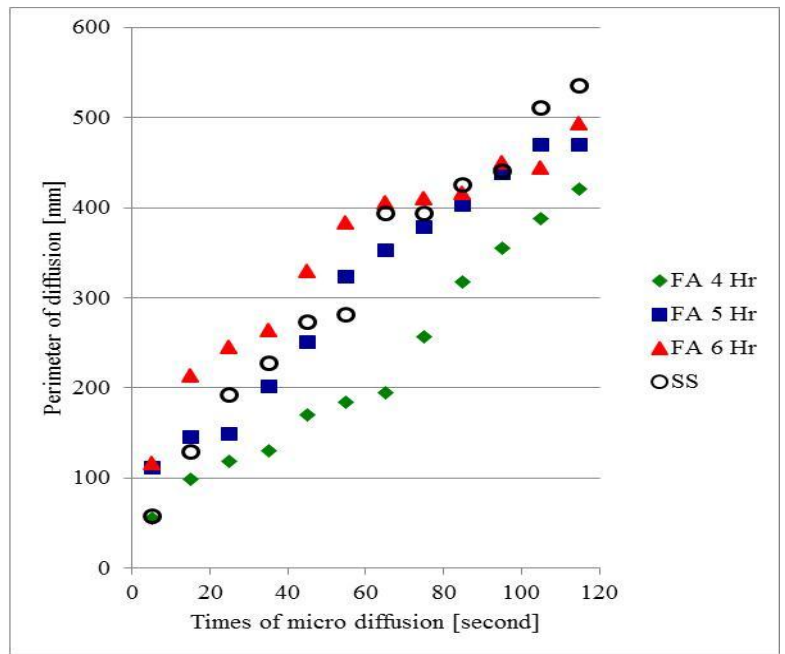
Fig. 6.The speed of interaction.

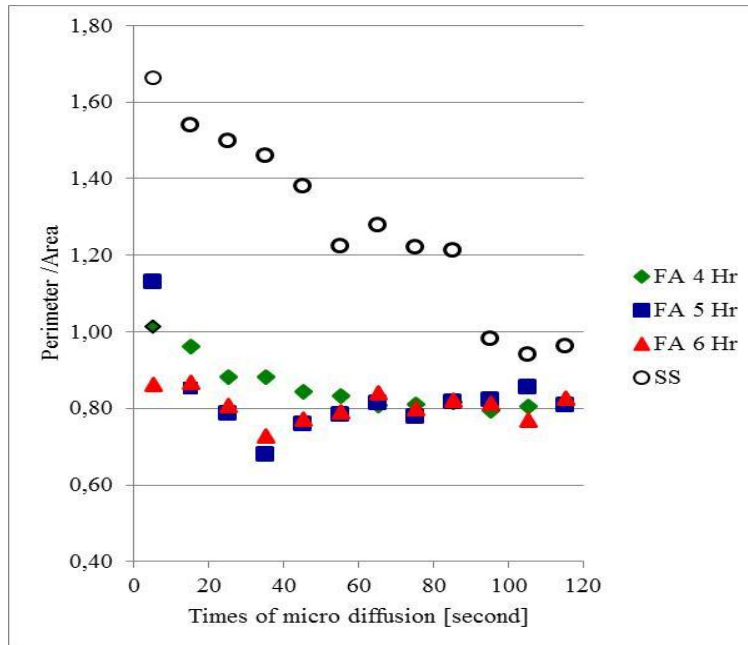Fig. 7. Perimeter of diffusion of FAMC and synthetic surfactant into ink structure

Fig. 8.Perimeter per area of diffusion.