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# Stability of skim latex suspension and rubber content recovery by microfiltration process: operating conditions and fouling characteristics

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#### ABSTRACT

To minimize the environmental impact of the latex industry and recover a large part of rejected skim latex, this research focused on the stabilization and concentration of skim latex suspension by chemical conditioning and microfiltration. The experiments were carried out in microfiltration stirred cell and cross-flow microfiltration unit to evaluate (1) suspension filterability and (2) critical permeate flux according to suspension conditioning. Critical permeate flux is defined as follows: when filtering feed suspension in cross-flow conditions during short time experiments, the trans-membrane pressure (TMP) increase induces firstly a linear evolution of permeate flux (*J*) before observing a progressive stabilization of *J*, even if there is a greater increase in TMP. The critical flux corresponds to the highest value of permeate flux obtained when the linear evolution J versus TMP is observed. Obtained results allowed the identification of critical filtering conditions, optimum chemical conditioning and incubation time to control membrane performances and fouling dynamics. The critical flux (J<sub>crit</sub>) depended on the dry rubber content DRC of the filtered latex suspension, and decreased from 48.3 to 7.4 l m<sup>2</sup>-h<sup>-1</sup> when DRC increased from 5% to 20%. Under this condition, it could be possible to achieve a concentrated skim latex suspension (about 20% DRC) with acceptable permeate flux. Nevertheless, during long periods of filtration, membrane fouling was observed even in sub-critical conditions, but it was easily reversed by a hydrodynamic method such as rinsing or flushing with tap water. The colloidal status of concentrated skim latex still occurred with an average volatile fatty acid (VFA) of 0.01% which means a good state of stability. To assess the efficiency of the membrane filtration, very low values of permeate turbidity were noticed, at about 25 NTU in subcritical condition and 50 NTU at critical condition (with a natural yellow color), in comparison with the initial turbidity of skim latex suspension, at about 60,000 NTU.

*Keywords:* Skim latex suspension; Microfiltration; Cross flow; Conditioning; Fouling; Rubber content recovery

#### 1. Introduction

Concentrated latex and skim rubber are the Thai rubber industry's main products. To produce concentrated latex, fresh latex is subjected to centrifugation to collect the rubber content but the smallest rubber particles are not completely extracted, giving the skim latex suspension a relatively significant rubber content (4-8% DRC), with high pH value (about 10-11 due to the addition of ammonia solution to avoid coagulation/ polymerization) and a large amount of proteins, sugars, lipids, carotenoids, inorganic and organic salts coming from natural rubber and process operation. Latex particle sizes in skim latex suspension are 2-10 times smaller than the average latex particle in concentrated latex. Rubber particles in skim latex can be recovered by using a coagulation process with acidification by adding sulfuric acid, but the recovery performance remains relatively poor and this operation generally has not been properly developed. The skim latex suspension is then discharged as serum wastewater after the skimming process is finished [1,2].

According to the sizes of particles and the capacity to flocculate in water when working in adapted pH range, microfiltration could be a pertinent solution to retain such particles and concentrate skim latex suspension in conformity with industrial valorization. In practice, the downstream latex industry requires a percentage of dry rubber content (%DRC) depending on what latex products they manufacture (latex gloves, latex pillows, latex condoms and latex thread) and processing application methods (dipping, foaming, casting or threading). The obtained concentrated latex suspensions are then diluted before being used as a raw material for the manufacture of each type of latex product.

There are few studies and reports on the recovery of such skim particles from latex industry wastewater. Nevertheless, this idea presents an actual challenge in terms of by-product recovery (without destroying quality) and of environmental protection. Indeed, if permeate, coming from microfiltration, still contains organic molecules, its concentration will be greatly lowered and it can be easier to use as substrate for bacterial and plankton culturing in aquaculture production or for biogas production instead of discharging it as concentrated serum wastewater. Therefore, the recovery of the smallest rubber particles from skim latex suspension and the reduction of organics in wastewater are of great interest to justify the choice of membrane filtration as a relevant means of separation and offer an alternative way to chemical conditioning [3,4]. However, for such concentrated suspensions the membrane fouling control appears as the main bottleneck, notably when the latex content increases with filtration time [5,6]. The association of filtration with a chemical conditioning of the skim suspension (e.g., the addition of detergents, with quaternary ammonium as cationic surfactant, or caustic solution of ammonia) then appears as a relevant step to provide latex particle stability in the serum content.

The objective of this research focused on the identification of appropriate operating conditions to recover the most concentrated skim latex suspension without developing a too high significant membrane fouling during filtration operation.

#### 2. Materials and methods

#### 2.1. Skim latex characteristics

Skim latex was taken from a concentrated latex factory in Songkhla province (South Thailand). The characteristics of feed samples were analyzed for important parameters such as pH, turbidity (turbidity meter: HACH model 2100N), total solids content (TSC: ASTM D1076: Section 8), dry rubber content (DRC: ASTM D1076: Section 9), volatile fatty acids (VFA: ASTM D1076: Sections 31–35) (indicating latex suspension stability during storage period, which should be less than 0.04%), total kjeldhal nitrogen (TKN: Macro – Kjeldahl Method), alkalinity (as NH<sub>3</sub>) (ASTM D1076: Section 10), total phosphorus (TP: Stannous Chloride Method), particle size distribution (MALVERN analyzer), and total protein with molecular weight size distribution of protein in serum by SDS page alkyl amine electrophoresis method [7,8].

#### 2.2. Chemical conditioning in Jar Test apparatus

The best conditions to stabilize and preserve the characteristics of the skim latex suspension were identified in Jar Test apparatus (PHIPPS & BIRD). The skim latex samples were conditioned by adding sodiumdodecyl (lauryl) sulfate (SDS) with a pH adjustment in the range of 7–8 by  $H_2SO_4$  or HCl addition (for five experiments as shown in Table 1). After adding selected chemical conditioning, namely, chemical type, sequence and doses, the influence of incubation time, meaning storage time before latex extraction/concentration, was studied for three values of 5, 8 and 11 d. All samples of conditioned skim latex suspension were stirred in rapid continuous mixing at 50°C, which was supposed to be a bad physical environment condition affecting the stability of skim latex. Then the best chemical conditioning pattern and incubation time were applied to prepare skim latex suspension prior to concentration by microfiltration.

#### 2.3. Microfiltration stirred cell unit and experiments

The filterability of the conditioned skim latex suspensions was first evaluated in a stirred cell microfiltration unit. Each test of skim latex suspension was operated

Sequence and type of chemical conditioning	Concentration of SDS	pН	Condition of mixing in the Jar Test
1. No chemical conditioning	-	10	Rapid mixing at 150
2. pH adjustment with $H_2SO_4$ followed by SDS addition	10 mg l <sup>-1</sup> (0.001% by weight)	7–8	rpm and slow mixing
3. SDS adding followed by pH adjustment with $H_2SO_4$	10 mg l <sup>-1</sup> (0.001% by weight)	7–8	at 40 rpm at room
4. SDS addition followed by pH adjustment with $H_2SO_4$	1000 mg l <sup>-1</sup> (1% by weight)	7–8	continued mixing at
5. SDS addition followed by pH adjustment with HCl	1000 mg l <sup>-1</sup> (1% by weight)	7–8	300 rpm at 50°C

Table 1 Chemical conditioning of skim latex suspension

with the best conditions defined in the Jar Test study. A schematic diagram of the experimental set-up is shown in Fig. 1.

The filtration was carried out under constant transmembrane pressure (TMP) (1 bar). The initial volume of suspension in the cell was 100 ml. Stirring was provided by a rotating impeller (300 rpm). To prevent the increase of the suspension concentration with time, filtration time was short (2 h) and the recovered permeate volume remained relatively small (20–25 ml.) with regard to the initial volume of suspension in the cell. The membrane characteristics are given in Table 2. The hydraulic resistance values obtained in this condition were calculated and compared with the results obtained with cross-flow microfiltration.



Fig. 1. Schematic diagram of microfiltration unit and membrane structure, (a) Stirred cell microfiltration unit, (b) SEM image of GSWP 04700 membrane ( $0.22 \ \mu m \ (x \ 10,000)$ .

Table 2
Microfiltration membrane characteristics

Characteristics	GSWP 04700 membrane
Туре	Flat-sheet
Membrane material	Mixed cellulose ester
Dimension (mm, diameter)	47
Filtration area (cm <sup>2</sup> )	11.9
Pore size (µm)	0.22
Porosity (%)	75
Total thickness (µm)	180
Water flux (20°C, 1 bar) (l h <sup>-1</sup> m <sup>-2</sup> )	10,800ª
Membrane resistance $R_{\rm m}$ (m <sup>-1</sup> )	$2.5 \times 10^{6a}$
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<sup>a</sup>Data from experiment tested.

#### 2.4. Crossflow microfiltration

To study the critical condition of filtration when concentrating the initial skim latex suspension of  $6.09 \pm 0.06\%$  DRC, semi continuous operations were carried out in a cross-flow microfiltration unit equipped with a ceramic membrane module. This unit was composed of a feed tank with a working volume of 25 l connected with a circulation piping system and a diaphragm pump (Fig. 2) that allowed imposing a defined crossflow velocity inside the membrane pipe. The tubular inorganic membrane characteristics are given in Table 3.



Fig. 2. Schematic diagram of a cross-flow microfiltration unit.

Table 3 Microfiltration membrane characteristics

Characteristics	
Туре	Tubular (19 channels)
Membrane material	Support material: α-Al <sub>2</sub> O <sub>3</sub>
	Membrane material: $\alpha$ -Al <sub>2</sub> O <sub>3</sub> /ZrO <sub>2</sub>
Pore size (µm)	0.22
Filtration area (m <sup>2</sup> )	0.24
Life time (years)	≈5
Membrane resistance $R_{\rm m}$ (m <sup>-1</sup> )	$1.34 \times 10^{11a}$
Water flux (20°C, 1 bar) ( $l h^{-1} m^{-2}$ )	250–270 <sup>a</sup>

<sup>a</sup>Data from experiment tested, membrane module from China.

Permeability of this membrane was measured at 20°C before the experiments using distilled water and the initial hydraulic resistance,  $R_{\rm m}$ , was calculated with an average of  $1.34 \times 10^{11}$  m<sup>-1</sup> according to Darcy's law.

#### 2.5. Experimental conditions in cross-flow microfiltration

All the experimental studies were conducted at a constant 3 m s<sup>-1</sup> cross-flow velocity and at a temperature of  $27 \pm 1^{\circ}$ C. Some conditioned skim latex suspensions were selected after Jar Test experiments for the determination of critical permeate flux  $(J_{crit})$  according to three DRC concentrations (5%, 10% and 20% DRC). During these experiments, the volumetric concentration factor (DRC concentrate/DRC permeate) was constant and equal to one due to a total recycling of permeate in the feed tank. The critical permeate flux values were investigated and identified by using the methodology reported by Gesan-Guiziou et al., Field et al. and Madaeni [5,9,10]. It was determined by systematic increasing of TMP step by step, 30 min at each TMP step before modifying TMP. At each TMP step, the corresponding permeate flux value (J) was noticed as soon as a constant value was reached. At the end of the experiment, the variation of J versus TMP was drawn. The TMP increase induced firstly a linear evolution of permeate flux (J) before observing a progressive stabilization of J even if there is a greater increase in TMP. The critical flux  $J_{crit}$  corresponds to the highest value of permeate flux obtained when the linear evolution J versus TMP is checked.

After the evaluation of  $J_{crit}$  for different DRC values, continuous experiments were carried out with a selected suspension at 5% DRC conditioned and at the best incubation time from topic 2.2. When operating, no permeate recycling in the feed tank was practiced. Then a progressive concentration of the suspension in the filtration loop occurred, characterized by the volumetric concentration factor. Filtration time was between 6 and 12 h. Filtration was operated by imposing the permeate flow successively at two values,  $J_{crit}$  and 50% of  $J_{crit}$ . The performances of filtration were evaluated through (1) the quality of permeate and retentate (by measuring % DRC and % TSC and protein content) and (2) membrane fouling dynamics by quantifying the evolution of the trans-membrane pressure (TMP) with time, and (3) fouling reversibility.

#### 2.6. Fouling characterization

The characteristics of fouling were analyzed and quantified by using conventional cleaning step procedures to determine resistance values at the end of each semi-batch operation. A cleaning in place procedure was operated step by step with a cross-flow velocity of 3 m s<sup>-1</sup> as follows: (1) rinsing and flushing the membrane with only tap water until no further skim latex was washed out, (2) flushing with sodium hydroxide solution 2 wt.% for 120 min, (3) backwashing with sodium hydroxide solution 2 wt. % for 120 min and (4) final backwashing with sodium hypochlorite solution 2 wt.% for 120 min. Membrane permeability was measured after each step of cleaning to identify and quantify the type and level of fouling that occurred.

#### 3. Results and discussion

#### 3.1. Skim latex characteristics

After the centrifugation process, the smallest latex particles still remained in serum fraction. The size of latex particles in skim latex was  $0.77 \pm 0.6 \mu$ m, values smaller than in raw latex suspension (0.05–5  $\mu$ m). The percentages of TSC and DRC were analyzed to identify the total solid content and dry rubber content, about  $9.89\% \pm 1.57\%$  and  $6.09\% \pm 0.06\%$ , respectively. The following chemical characteristics: COD, TKN and TP were respectively  $180,000 \pm 32,000, 1,400 \pm 900$  and  $60 \pm$ 35 mg l<sup>-1</sup>, pointing out the significant concentrations of pollutants in such suspensions if they are discharged as wastewater. The skim latex samples had pH values in a basic range  $(9.55 \pm 0.21)$  due to the addition of ammonia to preserve coagulation, even polymerization, of rubber content in serum. The protein concentration in the serum was close to 670 mg l<sup>-1</sup>. The Alkalinity of skim latex suspension was about 0.28% due to initial NH<sub>3</sub> conditioning of field latex. The VFA value indicated the stability of field latex (about  $0.0084\% \pm 0.002\%$ ). The physical characteristics of skim latex are given in Table 4.

#### 3.2. Effect of chemical conditioning on latex suspension stability

It appeared important to condition the suspensions to maintain rubber particles in colloid status in order to

Table 4 Skim latex characteristics

Parameter	Range	Average $\pm$ SD
pН	9.90–9.52	$9.55 \pm 0.21$
Turbidity (NTU)	63,000–54,100	$60,000 \pm 4242$
Total solids content (%)	11.01-6.33	$9.89 \pm 1.57$
Dry rubber content (%)	6.06-3.84	$6.09\pm0.06$
Alkalinity (as $NH_3$ ) (%)	0.29-0.26	$0.28 \pm 0.02$
Particle size (µm)	1.63-0.07	$0.77\pm0.56$
COD (mg l <sup>-1</sup> )	218,181–116,129	179,682 ± 31,891
TKN (mg l <sup>-1</sup> )	2744–714	$1372 \pm 930$
TP (mg l <sup>-1</sup> )	71.28–25.39	$61 \pm 34.75$
VFA (%)	0.01 - 0.007	$0.008 \pm 0.002$
Protein (mg l <sup>-1</sup> )	668–666	$667 \pm 1.46$

preserve the stability of skim latex suspension and control membrane fouling during microfiltration. The chemical conditioning was studied by adding sodiumdodecyl (lauryl) sulfate (SDS), related to pH adjustment in the range of 7-8 (by H<sub>2</sub>SO<sub>4</sub> or HCl adding) as shown in Table 1. It could maintain latex particles in colloid status. pH adjustment with H<sub>2</sub>SO<sub>4</sub> was followed by the addition of SDS, inducing a partial latex coagulation, while no chemical conditioning (only 2% ammonia from field latex) still presented a good colloidal phase. It was also found that the addition of 1000 mg l<sup>-1</sup> SDS followed by pH adjustment could extend the preservation life of skim latex suspension to more than 1 wk while at the low concentration of SDS, 10 mg l-1, skim latex suspension was destabilized and latex particles coagulated within 1-3 d. The influence of incubation time (storage time before latex extraction/concentration) was studied (5, 8 and 11 d). Fig. 3 shows the influence of incubation (storage) time on latex particle distribution size. 5 and 8 d storage induced latex particle sizes (respectively 0.951  $\pm 0.751 \,\mu\text{m}$  and  $1.018 \pm 0.805 \,\mu\text{m}$ ) were about double the size of when incubating time was 11 d ( $0.631 \pm 0.515 \,\mu$ m). It showed that lower incubation time had a positive effect on particle size due to the effect of the stable



Fig. 3. Effect of incubation time on latex particle size distribution (conditioning: 1000 mg SDS  $l^{-1}$  and pH adjustment): (A) = without conditioning, (B) = 5 d, (C) = 8 d and (D) = 11 d.

formation of micelles equilibrium when SDS could be attributed to the localized latex particles and structure, polarity and hydrophobicity of environment in micelles. Because of the interest in working with the largest particles when filtering (but without any significant latex precipitation), a storage time of 5 d was selected to concentrate conditioned skim latex suspension in ceramic membrane (*see* topic 3.4).

## 3.3. Effect of chemical conditioning on punctual latex filterability

Some filtration tests were carried out with the filtration lab cell unit to study the influence of latex chemical conditioning on suspension filterability. Results are illustrated in Fig. 4. When the addition of SDS was first operated before pH adjustment (whatever the SDS concentration), the evolution of permeate flux was close to or hardly higher than the evolution obtained with raw suspension (without conditioning). Compared to water membrane permeability (Table 2), hydraulic resistance appeared very high whatever the condition. The retention of skim latex particles onto the membrane surface (decrease of 98% turbidity in permeates) greatly reduced membrane permeability. Nevertheless, it could be observed that a constant permeate flux was acquired over time: then the hydraulic resistance quickly reached a constant value ( $4.25 \times 10^{12} \text{ m}^{-1}$  in comparison with the clean flat sheet membrane resistance  $8.83 \times 10^9$  m<sup>-1</sup>). When comparing this fouling resistance value obtained in stirred cell with the value found in cross-flow filtration,  $1.13 \times 10^{12} \,\mathrm{m^{-1}}$  at 50%  $J_{\rm crit}$  and  $2.26 \times 10^{12} \,\mathrm{m^{-1}}$  at  $J_{\rm crit}$ , it was found to be in the observed range. This observation let us conclude that fouling was controlled by imposed turbulence close to the membrane surface.

When the pH adjustment was made before SDS addition, the initial permeate flux appeared higher



Fig. 4. Flux evolutions with time for stirred cell microfiltration (SDS addition 10 mg  $l^{-1}$  and 1000 mg  $l^{-1}$  in the initial volume of skim latex suspension 100 ml).

than in other conditions, probably due to some initial coagulation due to the destabilization of some latex particles. Nevertheless, a quick decrease of permeate flux was observed reaching values close to those obtained in other conditions. The turbidity of permeate (serum) was decreased by about 98% from the initial values in raw skim latex suspension. It proves the great interest in microfiltration in terms of latex recovery and of course in terms of environmental impact if only the permeate represents wastewater minimization. The values of pH and TP were no different with and without the addition of SDS. Nevertheless, when the skim latex was conditioned by 1000 mg l<sup>-1</sup> SDS, the permeate quality showed higher values of turbidity (550 ± 346 NTU) and COD  $(35,000-55,000 \text{ mg } l^{-1})$  than when conditioned by 10 mg  $l^{-1}$ SDS (the permeate presented then a better quality with turbidity and COD, varying respectively in the range of 3-5 NTU and 14,000-18,000 mg l<sup>-1</sup>). This was probably due to the presence of excessive negative charges when SDS was added in excess, then latex particles (also with negative surface charge) and SDS pushed them away, and some latex particles crossed the membrane.

### 3.4. Concentration of skim latex suspension in a ceramic membrane unit

## 3.4.1. Critical conditions for concentrating skim latex suspension

Experiments were carried out to identify critical permeate flux when filtering conditioned skim latex suspension at different DRC concentrations [5,9,10]. The conditioning was defined as follows: 1000 mg l<sup>-1</sup> SDS and pH adjustment, 5 d of incubation time. The crossflow velocity in the membrane module was fixed at 3 m s<sup>-1</sup>. Fig. 5 and Table 5 present the results obtained. When filtering the two lowest concentrated skim latex



Fig. 5. Determination of critical conditions for 5, 10 and 20%DRC (skim latex suspension conditioning : 1% SDS and pH adjustment).

Table 5 Critical permeate flux ( $J_{crit}$ ) and sustainable operating transmembrane pressure when filtering conditioned skim latex suspension (1% SDS – pH adjustment – cross-flow velocity 3 m s<sup>-1</sup>)

Conditions	Critical flux $(J_{crit,} l m^{-2} h^{-1})$	Operating trans- membrane pressure (TMP, bars)
1. 5% DRC in skim latex with 1% SDS and pH adjustment	48.3	0.2
2. 10% DRC in skim latex with 1% SDS and pH adjustment	21.4	0.15
3. 20% DRC in skim latex with 1% SDS and pH adjustment	7.4	1.25

suspensions, evolutions of J vs. TMP were obtained: a first linear relation followed by a break in the curve corresponding to the critical permeate flux value which appeared depending on the latex concentration (48 l m<sup>-2</sup>-h<sup>-1</sup> and 21 l m<sup>-2</sup>-h<sup>-1</sup> for 5% and 10% DRC latex concentration, respectively). For both concentrations the break in the curve appeared in the same range of TMP (0.15-0.20 bar). When filtering the highest concentrated skim latex suspension (20% DRC), no permeate flux occurred when using low pressure driven conditions, the TMP must be higher than 0.75 bars to observe permeation. These results corresponded with the results reported by Konieczny and Bodzek, Novalic et al. and Gesan-Guiziou et al. [3-5]. These results show the great dependence between critical permeate flux and latex suspension concentration.

After non linear determination of permeate flux and TMP was detected, permeate flux decreased while TMP increased, or permeate flux increased again when higher pressure was increased (for example with 5% DRC, at pressure 0.5 bar flux was lower than 0.25 bar and increased again when pressure was 0.75). This phenomenon was also found in experiments with 10% and 20% DCR. It is a supercritical condition, caused by concentration polarization. Also, some of the latex particles accumulated as a non compact latex cake layer or dynamic membrane fouling on membrane surface, while cross-flow velocity at 3 m s<sup>-1</sup> made it possible to achieve certain permeate flux.

# 3.4.2. Continuous filtration: Identification of main fouling origin

In order to avoid membrane fouling especially from cake formation, and to concentrate skim latex suspension, cross-flow filtration mode was recommended to operate at  $J_{crit}$  and 0.5  $J_{crit}$  conditions, with a cross-flow

velocity of 3 m s<sup>-1</sup>, in order to reduce the energy input [4,11] for increasing latex concentration at around 20% DRC. Higher cross-flow velocity was still suggested in order to achieve a higher concentration of skim latex suspension when the average permeate flux was limited.

The chosen skim latex suspension was filtered continuously at  $J_{crit}$  and at 0.5  $J_{crit}$ . At the end of the operation, the resistance values to permeation were calculated and identified according to the described cleaning method steps as (i) reversible fouling (fouling due to particle accumulation and cake layer onto the membrane surface and the regeneration of permeability made possible by hydrodynamic technique) and (ii) irreversible fouling (pore blocking or adsorption and the membrane regeneration only made possible by using chemical reagents in cleaning solutions). The resistance values of the different fouling origins are shown in Figs 6 and 7.

When filtering the skim latex suspension at 0.5  $J_{crit'}$  the final resistance ( $R_{\rm f}$ ) appeared 10 times higher than the initial ceramic membrane resistance ( $1.34 \times 10^{11} \text{ m}^{-1}$ ). The results showed that irreversible fouling represented about 66% ( $7.52 \times 10^{11} \text{ m}^{-1}$ ) of final resistance. When operating at  $J_{crit'}$  the final resistance ( $R_{\rm f}$ ) value was 17 times more than the initial membrane resistance ( $R_{\rm m}$ ).



Fig. 6. Resistance values after each step of cleaning at the end of filtration at crossflow velocity  $3 \text{ m s}^{-1}(R_{\text{m}} = \text{initial membrane} \text{ resistance}, R_{\text{f}} = \text{final resistance}, R_{\text{f1}} = \text{fouling resistance after} \text{ rinsing and flushing with tap water}, R_{\text{f2}} = \text{fouling resistance} \text{ after flushing with 2 wt.% NaOH for 120 min, } R_{\text{f3}} = \text{fouling} \text{ resistance} \text{ after backwashing with 2 wt.% NaOH for 120 min, } R_{\text{f4}} = \text{fouling resistance after backwashing with 2 wt.% sodium hypochlorite for 120 min), (a) at 0.5 J_{\text{crit}} condition, (b) at J_{\text{crit}} condition.$ 



Fig. 7. Characteristic of membrane fouling after each step of cleaning at the end of filtration (cross-flow velocity 3 m s<sup>-1</sup>, Rrev = reversible fouling, Rirr = irreversible fouling).

Reversible fouling ( $R_{\rm fl}$ ) corresponded to a value of 2.02 × 10<sup>12</sup> m<sup>-1</sup>, about 90% of final resistance. The following observations were made regarding the results:

Although working at J<sub>crit</sub> and also in subcritical conditions, particle deposits remained effective when working over a long period. That is probably due to the large particle size distribution in skim latex suspension and the possible interactions between membrane surface and latex particles favorable to deposit development. That also means that the determination of critical flux obtained during short experiments represents the complex phenomena occurring in long term experiments.

Table 6

Characteristics of skim latex suspension and products after concentration

• The positive result is to show the important role of deposits, meaning it should be easy to control the fouling by periodic backwashing, for example [12].

Due to the cleaning methods in this study, as described in topic 2.6, membrane fouling after filtration was characterized at 0.5  $J_{crit}$  and  $J_{crit}$ . It was normal that the total duration time of cleaning was longer than the operation time. However, based on the system performance of concentrating skim latex suspension operated at a cross-flow velocity of 3 m s<sup>-1</sup> and  $J_{crit}$ , most possible fouling is of the reversible type, which is easy to control by using an intermittent mode of high cross-flow velocity raised to 4 m s<sup>-1</sup>.

### 3.4.3. Concentrated skim latex and serum quality from cross-flow microfiltration

Some criteria characterizing the quality of the retentate and permeate at the end of the operation are given in Table 6 when filtering at 0.5  $J_{crit}$  and  $J_{crit}$ . The percentage of TSC in the initial conditioned skim latex suspension was two times higher than the percentage of DRC (because TSC is composed of rubber and non rubber content). The concentration of conditioned skim latex suspension by filtration increased the percentage of DRC in retentate while non rubber content was additionally present in permeate. The comparison of retentate characteristics between the filtration at  $J_{\rm crit}$  and 0.5  $J_{\rm crit}$  also points out the role of deposits for intensifying the retention of particles (the proportions of TSC and DRC were higher when the condition of filtration were more severe). These results also point out the possibility of concentrating the skim latex suspension until it reaches a volumetric concentration factor close to three for TSC and five for DRC.

Parameters	Skim latex	Permeate (serum)		Retentate (concentrated skim latex)	
		at $0.5 J_{\rm crit}$	J <sub>crit</sub>	at 50% $J_{\rm crit}$	$J_{\rm crit}$
pH	8.14 <sup>a</sup>	8.44	8.88	8.47	8.9
Turbidity (NTU)	60.00	22.85	47.40	_	_
Total solids content (%)	8.17	_	_	16.89	23.54
Dry rubber content (%)	4.05	_	_	12.84	19.50
VFA (%)	0.01	_	_	0.03	0.02
TKN (mg l <sup>-1</sup> )	4368	3444	3340	4676	4284
Viscosity (C p)	3.5	_	_	4.5	7.5
Protein (mg l <sup>-1</sup> )	667	530	475	_	_
Alkalinity (as NH <sub>3</sub> ) (%)	0.29	_	_	0.04	0.07
TP (mg l <sup>-1</sup> )	-	2.53	1.25	_	-

<sup>a</sup>After pH adjustment in the range of 8-8.5 with HCl.

The VFA value (<0.04%) gives a good indication of the colloidal status. For both filtration conditions the colloidal status of concentrated skim latex was in a range of 0.02–0.03% of VFA. The turbidity in permeate appeared respectively close to 25 NTU and 50 NTU for the two conditions. The permeate serum was clear and had a natural yellow color. The organic substances in skim latex suspension in term of COD, rubber and non rubber contents, were on average  $179,682 \pm 31,891 \text{ mg } l^{-1}$ . The removal efficiency in terms of COD by microfiltration was in the high range of 90% with COD values of about 16,000  $\pm$  2000 mg l<sup>-1</sup> in the permeate samples. The protein content in permeate serum was about 530 mg l-1 (the initial protein contents in skim latex are given in Table 6). The fraction of protein content in permeate was characterized using SDS page alkyl amine electrophoresis method. It was found that most of the proteins presented a molecular weight distribution (MW) in a low range between 14 and 36 KDa that corresponded to Glucanase, 29 KDa of Hevamin or Chitin, 20 KDa of small rubber particle protein or Prohevein, 14.2 KDa of rubber elongation factor protein and MW of protein lower than 14.2 KDa of Hevein.

The concentrated latex (in terms of %DRC) obtained from this preliminary study can be utilized as raw material to mix with other chemical compounds for latex adhesive production. The permeate or serum contained a large amount of proteins, TKN, organic (sugars and lipids) and inorganic salts, which can be utilized as substrate for bacterial and plankton culturing in aquaculture production or for biogas production instead of being discharged as wastewater. Using this concept, wastewater treatment plants and the environmental impact of the latex industry can be significantly reduced.

#### 4. Conclusions

Chemical conditioning with SDS (before pH adjustment) of skim latex suspension was effective to stabilize latex particles in some colloid dispersion states. When filtering such suspensions in specific stirring, a suspension conditioning with SDS (1000 mg l-1) associated with a pH adjustment (in the range of 8-8.5) showed the best results in terms of permeate flux control, hydraulic resistance reversibility, concentrate and permeate quality. The incubation time influenced latex particle size and suspension stability, and also depended on chemical conditioning. The initial concentration of the latex suspension and/or the imposed volumetric concentration factor directly influenced the dynamics of filtration. Results show the possibility of obtaining, by cross-flow filtration, a stable final skim latex suspension with a DRC close to 20% using an initial suspension where DRC was 4%.

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#### Symbols

DRC	 dry rubber content
$R_{\rm irr}$	 irreversible resistance
R	 reversible resistance
SDS	 sodium dodecyl (lauryl) sulfate
TKN	 total kjeldahl nitrogen
TMP	 transmembrane pressure
TP	 total phosphorus
TSC	 total solids content
VFA	 volatile fatty acids

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