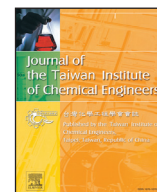




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## Optimization of pulp fibre removal by flotation using colloidal gas aphanes generated from a natural surfactant

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### ABSTRACT

Colloidal gas aphanes (CGAs) are a system of highly stable micro bubbles in colloidal state. In this study, the CGAs prepared from a natural surfactant saponin, extracted from the fruit pericarp of *Sapindus mukorossi* or soapnut plant, was utilized for the recovery of pulp fibres from paper machine backwater in a flotation column. The performance of soapnut CGAs was compared with that of CGAs generated from cationic, anionic and non-ionic surfactants. Performance optimization of soapnut CGAs was undertaken using central composite design (CCD). CGAs characterization showed that soapnut surfactant produced the most stable CGAs. Under various CGAs sparging rate, pH and flow rate of wastewater, soapnut CGAs performed best by removing up to 60% total suspended solids (TSS) from paper machine effluent as compared to 50%, 37% and 30% TSS removal by cationic, anionic and non-ionic surfactants respectively. Optimized TSS removal of 76% was attained through CCD at soapnut CGAs sparging rate of 0.013 L/min, wastewater flow rate of 16 L/min and pH of 7.5. Treatment of effluent using natural surfactant CGAs is a cost effective and green process which can be replicated in industries.

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### 1. Introduction

Colloidal gas aphanes (CGAs) are a system of microbubbles mostly above 25  $\mu\text{m}$  diameter and classified as kugelschaums or “ball foams”, first described by Sebba [1]. CGAs can be generated by high speed stirring of the surfactant solution ( $\sim 6000$  rpm), whereby air is entrapped and microbubbles are formed. The CGAs are suitable for process applications due to their ability to adsorb particles at microbubble interfaces, their large interfacial area and their stability during transport for enhanced mass transfer [1]. Earlier, CGAs had been applied for the separation of fine particles through flotation process in a column [2–6]. Froth flotation process using foams has several advantages over other processes particularly in the removal of fine particles, which do not have practical settling rates under gravity, and in the separation of light particles which tend to float. Foam, however is hard to be pumped as it loses its characteristics due to its rheology while CGAs can be easily pumped.

Paper production is a highly water intensive process and consequently generates large quantity of waste comprising fine pulp fibres which escape through the fine wire mesh on which paper is formed [7,8]. Recently, chitosan has been used in dissolved air flotation (DAF)

process to recover pulp fibres [9]. However, flotation of paper fibres by CGAs generated from saponin has never been undertaken and this is completely different from DAF process. The nature and characteristics of the CGAs are influenced by the type and concentration of the surfactants, and the ionic nature of the surfactant has been shown to be very important for the functioning of the CGAs.

This work aims to explore the efficiency and optimize the performance of CGAs generated from natural surfactant saponin extracted from soapnut fruit pericarp for TSS removal from paper mill effluent. The CGAs generated by soapnut was compared with other common synthetic surfactants, based on stability and liquid drainage time. The generated CGAs were applied for the recovery of pulp fibres from paper mill effluent and the operating parameters for saponin were optimized using central composite design.

### 2. Materials and methods

#### 2.1. Surfactants

Four surfactants were used in this study, of which one is of plant origin saponin and three were synthetic. Saponin is a natural surfactant traditionally used as an environmental friendly detergent [10] and is non-ionic at pH 3.5 and displays slightly anionic character with increasing pH [11]. It was extracted from the

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soapnut fruit pericarp by water [12] and the extract contained about 65% saponin as determined by UV–vis spectrophotometer [13]. The synthetic surfactants used were sodium dodecyl sulphate (SDS), Triton-X100 and cetyltrimethylammonium bromide (CTAB). Soapnut solution was used at a concentration of 0.5% (w/v). The synthetic surfactants were used at concentrations of 7 mM for SDS, 1 mM for Triton-X100 and 1 mM for CTAB. The CGAs generated by the surfactants were characterized by liquid drainage, air hold-up and half-life ( $t_{1/2}$ ) as proposed by Zhang et al. [14].

## 2.2. Paper mill effluent preparation

Synthetic paper machine backwater effluent stock solution was prepared in the laboratory by mixing 2 g of ordinary tissue paper in 1 L distilled water to prepare the stock solution in order to maintain uniformity throughout the extensive batch experiments [15]. The stock solution was diluted 10 times to mimic paper machine backwater fibre concentration. The resultant wastewater had 200 mg/L of paper fibre concentration. The pH of the wastewater is near neutral ( $\cong 6.5$ ) and the turbidity of the effluent is 80.6 NTU. No chemicals were added to the diluted slurry and it was prepared fresh for each set of experiments to prevent bacterial degradation.

## 2.3. Generation and characterization of CGAs

Colloidal gas aphanes were generated from surfactant using a homogenizer (IKA T 25 basic ULTRA-TURRAX®). The surfactant solutions were stirred at high speed (6500 rpm), starting with 500 mL of surfactant solution, until a constant volume of white creamy CGAs were produced in 6 min. These CGAs once produced, were kept dispersed under low stirring conditions at around 1000 rpm by a magnetic stirrer and were pumped into the flotation column using a peristaltic pump (Sastec BT 100-2J) at different sparging rates of 0.007, 0.010, 0.013, 0.016 and 0.018 L/min.

## 2.4. Flotation of fibres using flotation columns

In order to remove the pulp fibres by flotation, the effluent and CGAs were passed in counter-current direction. The CGA bubbles rise up slowly due to their small sizes. The fine paper fibres coming down with the wastewater from top of the column come in contact with the bubbles rising upwards and are carried upwards by the bubbles and are removed with the foamate. The flotation column is made of Perspex glass, 0.05 m in diameter and 1 m in height. The CGAs inlet was at 0.06 m from the base of the column and an outlet at the base of the column for the tailings. A conical diffuser was positioned at the base, just above the CGAs inlet to achieve a uniform distribution of aphanes. The height of liquid in the column was maintained by constantly pumping the wastewater from the top of the column at a constant flow rate. The inlet for the wastewater was at 0.665 m above the base of the column. The foamate and the entrapped particulate matters were collected from the top of the column. The wastewater was initially poured into the column until it reached just above the feed inlet. Then the CGAs were pumped from bottom of the column. The experimental scheme is shown in Fig. 1. Each set of experiments was run for 80 min and samples were collected every 10 min. The system required 30 min to stabilize and the data after the stabilization phase is presented here.

## 2.5. Optimization experiments

Central composite design (CCD) having five levels effective for the estimation of parameters in a second order model was developed by Box–Hunter [16]. A second-degree polynomial equation is used to

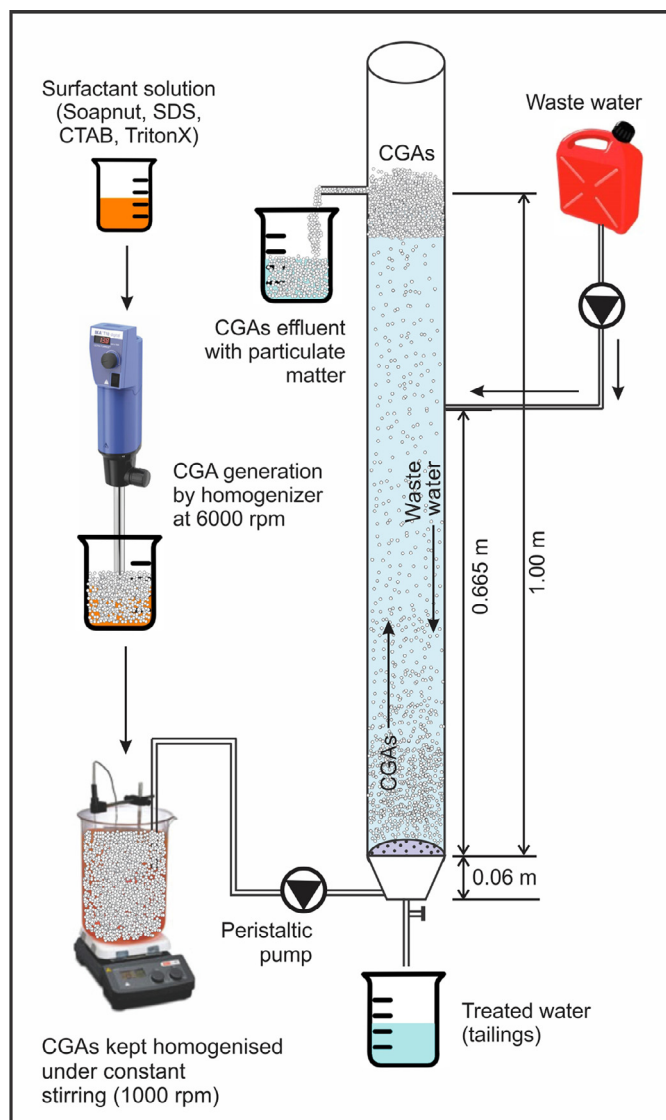


Fig. 1. The scheme of the experiment.

explain the behaviour of the system, as shown in Eq. (1):

$$y = \beta_0 + \sum_{i=1}^k \beta_i x_i + \sum_{i=1}^k \beta_{ii} x_i^2 + \sum_{i \geq j}^k \sum_{i=1}^k \beta_{ij} x_i x_j \quad (1)$$

where,  $y$  = predicted response,  $\beta_0$  = offset term,  $\beta_i$  = linear effect,  $\beta_{ii}$  = squared effect,  $\beta_{ij}$  = interaction effect.

Several factors that can influence the removal of TSS by CGAs flotation were taken as variables and their coded and actual values are listed in Table 1. All the experimental designs and optimization were performed using Design Expert 7 software.

Table 1  
Actual values of variables for the coded values.

Variables	Actual values for the coded values				
	$-\alpha$	-1	0	+1	$+\alpha$
CGA sparging rate (L/min) (A)	0.00725	0.010	0.013	0.016	0.01805
Wastewater flow rate (L/min) (B)	0.00725	0.010	0.013	0.016	0.01805
pH (C)	5.15	6	7.25	8.5	9.35

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