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# Removal of thermophilic spores from gum Arabic streams using ceramic alumina microfiltration membranes

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ABSTRACT

The microfiltration (MF) of high solids content gum Arabic solutions (15 wt%) inoculated with *Bacillus mycoides* spores ($10^5$ CFU $ml^{-1}$) was carried out using Membralox tubular ceramic membranes with a nominal pore size of 0.8 μm. Consistent permeate fluxes were achievable over multiple fouling and cleaning cycles, while giving low rejection of solids and high rejection of spores (after ten cycles, a permeate flux of 42.9 L $m^{-2}$ $h^{-1}$, a solids retention of 19.8%, and a spore rejection of 5.0 log orders were achieved). Although fouling during filtration was severe, permeate fluxes could be restored to a satisfactory condition after cleaning with 0.5 wt% NaOH solution containing 200 ppm NaOCl. Results were described by a two species first order removal model, whereby one species was removed quickly by cleaning and the other was more difficult to remove. The optimum cleaning time for NaOH + NaOCl solutions at 60 °C was found to be ca. 20 min. Subsequent citric acid cleans had a negative effect upon restoring permeate flux.


## 1. Introduction

Gum Arabic (GA) is the dried exudation obtained from various species of *Acacia* trees of the leguminosae family. About 500 species of *Acacia* are distributed across tropical and subtropical areas of Africa, India, Australia, Central America and southwest North America, but only a few are commercially important. Most production occurs in the 'gum belt' proper of central and western Sudan where the trees *Acacia Senegal* grow in sandy soils under water scarce conditions. GA is the country's second largest export and Sudan provides 85% of the world supply of GA, which is predominantly used in the food and beverage industry for its emulsifying and stabilising properties.

Gum Arabic is a slightly acidic complex polysaccharide that consists of three main components; (i) arabinogalactan (AG) with a molar mass of approximately 280 kg $mol^{-1}$ that makes up ≈88 wt%, (ii) arabinogalactan-protein (AGP) complex which accounts for ≈10 wt%, and has a molar mass of 1500 kg $mol^{-1}$, (iii) glycoprotein (GI) which makes up the final ≈2 wt% and has a molar mass of 250 kg $mol^{-1}$. The AGP complex is the active component responsible for GA's emulsifying and stabilising properties (Fauconnier et al., 2000; Manning and Bird, 2015; Manning et al., 2016).

It is well known that conditions in which GA is grown and collected are susceptible to contamination by bacterial spores such as *Bacillus cereus* (Farag Zaied et al., 2007); a known food-poisoning bacterium which is resilient to thermal pasteurisation. Removal of such spores is thus difficult by heat treatment alone given the thermo-resistant properties and resulting degradation of product quality with excessive temperatures (Decloux et al., 1996). Therefore the use of a filtration method to act as a cold pasteurisation technique is an attractive alternative.

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In this study, microfiltration was used as a method of cold pasteurisation. Reconstituted spray-dried gum solutions were inoculated with a microbial load of *Bacillus mycoides* spores (maximum diameter $1.53 \pm 0.18\,\mu m$ (Head and Bird, 2013b) as an analogue to *B. cereus*. The gum solutions were then micro-filtered over multiple fouling and cleaning cycles with the objective of achieving high permeate flux, high gum solids transmission and high spore retention over a period of time, and measuring the effect of membrane ageing on filtration performance. The overall aim of the experiments was to develop an industrially relevant protocol to modify industrial practice. Feeds, processing conditions and cleaning agents were all selected according to previous industrial practice.

The removal of bacteria from food and dairy products using microfiltration is a well-established and proven technology (Pafylias et al., 1996; Tomasula et al., 2011). However, typical dairy microfiltration processes treat low solids content aqueous streams; skimmed milk has a protein content of approximately 3.5 wt% (Piry et al., 2008). Higher solids content streams such as those used in the processing of milk protein isolate are more difficult to filter effectively due to higher viscosities and higher rates of membrane fouling leading to low flux and solids transmission (Head and Bird, 2013a,b). Similar difficulties were expected with high solids content GA feeds.

## 2. Materials

### 2.1. Experimental apparatus

The experiments were carried out on a laboratory scale cross-flow microfiltration apparatus as described by Head and Bird (2013b). The apparatus consisted of two circulation loops. A centrifugal pump circulated feed from the tank (20 L) through a heater to raise the feed to the desired temperature, and then by adjusting control valves the feed was diverted to the retentate circulation loop. The retentate was partially recycled through a cooler to the retentate circulation pump. Trans-membrane pressure, temperature and cross-flow velocity were measured by an arrangement of pressure transducers, a thermocouple (Cole-Parmer) and electromagnetic flow metre (MAG Magflo 1100). Permeate flux measurements were logged automatically to a computer.

### 2.2. Membranes

The membrane tested was a Pall Membralox™ (Pall, USA) tubular ceramic membrane. The membrane had a nominal pore size of 0.8 μm, 19 cylindrical channels, 1020 mm long and 4 mm in diameter giving an active membrane area on each membrane of $0.24\,m^2$. However, to achieve high cross flow velocities on the same experimental apparatus, only 3 channels were used by blocking off 16 channels using stainless steel backed plugs and gaskets, giving an active membrane area of $0.04\,m^2$. By pure water flux measurements, this was shown to a suitable method of increasing cross-flow velocity for laboratory scale measurements without increasing the size of the related apparatus.

### 2.3. Gum Arabic fouling suspension

The gum Arabic used was supplied by *Kerry group plc.* as a spray-dried powder. For each experiment, 16 kg of solution was made up the previous day by mixing the gum powder with reverse osmosis treated water (measured conductivity of $8.0\,\mu S\,cm^{-1}$) at room temperature and stirred continuously for 2 h. The solution was left overnight at room temperature to ensure complete gum dissolution. When diluted with holding water in the rig a feed of 15 wt% GA solids resulted.

### 2.4. B. mycoides spore inoculation

The method used to prepare a spore solution of *B. mycoides* is described by Seale et al. (2008). Ten millilitres of the spore solution was pipetted to the appropriate feeds before experiments to give a retentate microbial load of ca. $10^5$ CFU $ml^{-1}$. The spores are ellipsoid in shape with a width of $0.9 \pm 0.11\,\mu m$ and length of $1.53 \pm 0.18\,\mu m$ (Head and Bird, 2013b).

### 2.5. Cleaning reagents

Three cleaning reagents were used. The first chemical cleaning stage used a solution of 0.5% w/w sodium hydroxide (NaOH) with 200 ppm sodium hypochlorite (NaOCl) added. The NaOH concentration chosen was that determined by Bird and Bartlett (1995) to be the most effective for proteinaceous deposit removal. The presence of NaOCl was included for two reasons, (i) It is a widely used and effective sanitiser even at low concentrations (Young and Setlow, 2003) and (ii) NaOH solutions alone proved to be ineffective at restoring acceptable permeate flux after gum Arabic fouling. It was found that adding 200 ppm NaOCl was effective in increasing flux recovery. In the microfiltration of rough beer Gan et al. (1999) demonstrated that combining caustic and oxidation cleans was more effective than running them in two separate stages. Gan et al. (1999) also found a flux recovery associated with an acid clean following alkali cleaning of a ceramic membranes. Likewise the second cleaning stage in these experiments used 0.1% w/w citric acid solution.

## 3. Methods

Each cycle consisted of seven stages; Initial pure water flux, fouling, a first rinse, NaOH + NaOCl cleaning, second rinse, citric acid cleaning and then a final rinse. Each cycle completed a full fouling and cleaning of the membrane, with the first of 10 successive cycles was carried out on a pristine membrane. The full conditions of each stage are given in Table 1. A typical profile of total resistance over one fouling and cleaning cycle can be observed in Fig. 1. Resistance increases during fouling for the first 60 min, with subsequent rinsing and cleaning stages removing fouling and lowering resistances (although the apparent resistance during resistance is higher than that seen during fouling – as discussed in Section 4).

### 3.1. Initial pure water flux (PWF)

Resistance to permeate flux could be measured at each stage of the cycle. Initial resistance by the pristine membrane ($R_{m1}$) was measured in the first stage of cycle one by measuring the initial pure water flux and using Eq. (1):

$$J = \frac{TMP}{\mu R_m} \tag{1}$$

In the same way, the resistance of the cleaned membrane ($R_n$) was calculated by taking the flux $J_n$ and substituting $R_m$ with $R_n$.