Pilot plant scale extraction of alginates from *Macrocystis pyrifera*. 2. Studies on extraction conditions and methods of separating the alkaline-insoluble residue

Gustavo Hernández-Carmona[1,2,3*], Dennis J. McHugh[4] & Francisco López-Gutiérrez[1]
[1] *Centro Interdisciplinario de Ciencias Marinas. Ap. Postal 592, La Paz, B.C.S., México*
[2] *Centro de Investigaciones Biológicas del Noroeste. S.C. Ap. Postal 128, La Paz, B.C.S., México*
[3] *Becario COFAA-IPN, PEDD-IPN and CONACYT*
[4] *School of Chemistry, University College, University of New South Wales, Australian Defence Force Academy, Canberra ACT 2600, Australia*

(* Author for correspondence: fax: +52-1-122-53-22; e-mail: gcarmona@balandra.uabcs.mx)

Abstract

The effect of temperature (70, 80, 90 °C) and time (1–9 h) during the alkaline extraction step on alginate yield and quality were studied. The alginate yield increased with time and maximum yield was obtained after 3.5 h treatment, ranging from 19.4% at 70 °C to 21.9% at 90 °C. The viscosity of the alginate produced was inversely correlated with the temperature and time. At 70 °C the slope of the curve was almost zero (753 to 923 mPa s); at 90 °C the viscosity loss was 154 mPa s per hour during the first two hours, reducing from 523 to 86 mPa s after 5 h; 80 °C yielded values between those for 70 °C and 90 °C. The best conditions for alkaline extraction were using pH 10 at 80 °C for two hours. The curves obtained gave useful information for controlling the viscosity of the alginate during production. It was found that viscosity of the paste formed during alkaline extraction ('process viscosity') was the best parameter to determine the reaction rate during extraction. Alginate yield increased during filtration time from 17.6% to 23.7% after 55 min at 70 °C. In this step the viscosity of the alginate obtained remained almost constant (522–610 mPa s), indicating no degradation of the products during filtration. The best dilution to filter the alginate extract was obtained at 45 mPa s. Diatomaceous earth (Celite) and expanded lava (Perlite) were tested as filter aids. Expanded lava was the best filter aid, using 1 kg per kilogram of alginate produced. Three methods were studied to separate the alkaline-insoluble residues after extraction: filtration, centrifugation, flocculation, and combinations of them. The best system found was filtration with flocculant in a rotary vacuum filter, with a knife advance of 0.1 mm every 3.5 seconds and drum rotation of 2 rpm, yielding an average filtration flow rate of 10.5 L min^{-1}.

Introduction

Alginate is a linear glycuronan of (1,4)-linked α-L guluronate (G) and β-D mannuronate (M) residues arranged in a non-regular blockwise pattern along the chain (i.e., it is not composed of simple oligosaccharide repeating units) (Grasdalen et al., 1981). It is a hydrocolloid with many applications in the food, pharmaceutical, cosmetic and biotechnology indus-

tries, etc. as a gelling agent, thickener or stabilizing and emulsifying agent (Istini et al., 1994).

Many reports and patents concerning the production of alginate have been published, however details of the successful commercial processes have not been made known (McNeely & Pettitt, 1973). The Clark & Green (1936) and Le Gloahec & Herter (1938) patents appear to be very detailed, but they obscure more

than they reveal, a commonplace problem with patents (Booth, 1975). Hernández-Carmona et al. (1999) described the use of pre-treatment with formaldehyde and one acid washing at pH 4 as the best treatment to obtain a product with high viscosity and good yield, revealing that ion exchange Ca^{2+}/H^+ was not as important as it had been described in previous papers. In this paper we describe the best parameters for the next three steps of the process: extraction, dilution and separation of residual insoluble algal material.

The purpose of the extraction step is to convert the alginate to the soluble form of sodium alginate so as to remove it from the algae. Little and fragmentary information exists about this step. Green (1936) patented a process without heating during the alkaline extraction and obtained a high viscosity alginate. However, the value of producing a very high viscosity alginate is debatable, because during storage this product is more prone to breakdown and loss of viscosity than a medium viscosity alginate (McHugh, 1987). Le Gloahec & Herter (1938) described the use of alkaline treatment with 4% sodium carbonate solution, for 2 h at 40 °C. Bescond (1948) suggested 5–7% sodium carbonate solution to digest the algae during 12 h. Haug (1964) recommended a pH below 7, with 50 parts of sodium hydroxide solution per one part of algae. Lukachyov and Pochkalov (1965) proposed the use of boiling sodium carbonate but they did not describe the treatment time. Zvered et al. (1969) used 1–2% sodium carbonate during 1–2 h at 27 °C. Secconi (1967) proposed treatment for 5–24 h with a basic solution of ammonia, potassium or sodium salts in the proportion of 5–7 kg of basic solution per 100 kg of algae; he did not specify the concentration of the basic solution but the paste obtained had a pH between 5 and 7.5. Baranov et al. (1967) used 12–15% sodium carbonate plus 5% sodium phosphate at 45 °C for 30 min. McHugh (1987) proposed extraction with 1.5% sodium carbonate solution for 1–2 h at 50–60 °C. Hernández-Carmona et al. (Pat. Pending) and Arvizu-Higuera et al. (1996), proposed extraction at pH 10 for 2 h.

The extraction step can be used to control the viscosity of the final product. Higher temperatures and longer extraction times lead to breakdown of uronic acid chains and consequent lower viscosities for the sodium alginate (McHugh, 1987). However none of the above authors give details of the degree of depolymerization (degradation) under different extraction conditions, nor if alginate is degraded during the filtration step. Therefore the first objective of this work

was to determine the effect, on the alginate yield and viscosity, of temperature and time during the extraction step at pilot plant level and of time during the filtration step at constant temperature (70 °C).

After the alkaline extraction, the dissolved sodium alginate must be separated from the alkali-insoluble seaweed tissue. The extract is diluted, to produce a suitable viscosity for filtration. Some of the alkali-insoluble material can be removed by centrifugation or by a flotation process, but for food grade alginates, a filtration step is necessary in all cases (Green, 1936; McHugh, 1987). No data has been published about the factors affecting the extent and speed of particle separation from the alginate extract, factors such as alginate concentration in solution, the type of filter aid used or the system used: filtration, centrifugation, flotation or a combination of these. Therefore, the second objective of this work was to determine the best equipment and parameters to separate alkali-insoluble residual algal tissue from the diluted alginate extract.

Materials and methods

Effect of temperature and time during alkaline extraction

Ten kg of the dried and milled alga *Macrocystis pyrifera* (L.) C. Agardh, collected from Bahía Tortugas, Baja California Sur, México, were placed in a tank to rehydrate overnight with 90 L of 0.1% formalin solution. The residual solution was drained off and the alga was washed with 100 L of hydrochloric acid at pH 4 in the same tank for 15 min with constant agitation (Hernández-Carmona et al., 1999). The alga was transferred to an extraction tank containing 166 L of water, the pH was adjusted to 10 using powdered sodium carbonate and the temperature was changed for each experimental treatment to: 70 °C, 80 °C and 90 °C. After the temperature in the extraction tank reached the experimental temperature, samples of 300 mL were taken from the extraction paste every 30 min up to 9 h (18 samples). The viscosity of the samples was measured in the laboratory using a Brookfield viscometer. This viscosity was called 'process viscosity', it is the viscosity of the undiluted alginate extract and includes the effect of the alkali-insoluble algal material in the paste. This viscosity was used to determine the rate of extraction.

An additional ten samples (each 165 mL) were taken from the extraction tank every 30 min. These

were diluted to 600 mL with hot water and filtered in a vacuum filter using filter paper UF50 and diatomaceous earth as filter aid. The filtrate was adjusted to 70 °C and the viscosity was measured in a Brookfield viscometer. This viscosity was called 'filtrate viscosity' and was used as another indication of the extraction rate; this filtrate viscosity avoids any possible interference from the presence of insoluble particles which are present in the process viscosity samples.

Sodium alginate was precipitated from 500 mL of each filtered sample by the addition of ethanol (500 mL). The product obtained was dried at 50 °C and weighed to calculate the yield. A 1% solution was prepared to measure the viscosity of the alginate, using the methods described in Hernández-Carmona et al. (1999). In all viscosity determinations, the solution was measured as it was (viscosity 1) and again after adding sodium hexametaphosphate (0.5 g per 100 mL of alginate solution) (viscosity 2). Viscosity 2 was used to determine the degree of depolymerization of the alginate caused by the time and temperature of extraction.

Effect of filtration time on the filtrate viscosity and alginate yield and viscosity

The paste of the alginate obtained after extraction was diluted with hot water (70 °C) to 600 L, and constant stirring was maintained. One sample (500 mL) from the dilution tank was taken to the laboratory and filtered. The viscosity of the filtrate was measured and then the alginate was precipitated with ethanol so that its yield and viscosity could be measured at time zero (before start of filtration). The rest of the diluted extract in the tank was filtered in a small industrial rotary vacuum filter.

This filter consists of a hollow cloth-covered drum, rotating on a hollow shaft, while partially immersed in a pan. The shaft is connected to a vacuum pump. The filter is precoated by feeding a suspension of filter aid in water into the pan. The vacuum pulls the filter aid onto the cloth, building up a layer of filter aid. The slurry that is to be filtered is fed into the pan, and is pulled through the filter aid by the vacuum and out via the hollow shaft. As the slurry is pulled through this layer of filter aid, the fine solid particles of the slurry are deposited on the surface. A knife blade is arranged to move slowly and automatically into the layer of filter aid, shaving off a small amount of it as the drum rotates, leaving a clean surface for rapid filtration (modified from Alar, 1991).

In this experiment the filter was covered with 14-m layer of filter aid (Celite Hyflo Super-Cel, a diatomaceous earth), the drum rotation was 2 rpm and the knife advanced 0.1 mm every 3.5 s. During filtration a temperature of 70 °C was maintained in the dilution tank. Samples of 500 mL of the clear liquor were taken every 8 min and at the end of filtration one sample was taken of the total mixed filtrate. The viscosity of each sample was measured (filtrate viscosity). These samples were treated with 500 mL ethanol and the precipitated sodium alginate was dried. The yield and viscosity of the alginate were determined using the methods described above.

Effect of alkaline extract dilution on the filtration rate

After alkaline extraction using the optimum conditions (80 °C, 2 h), the paste was diluted to the following volumes: 300, 350, 400, 450, 500, 550, 600, 650 and 700 L. A sample of 300 mL was taken from the paste and from each dilution and the process viscosities were measured at 70 °C. In order to isolate the sodium alginate and determine its concentration in each dilution, the paste and the samples diluted to 300, 400, 500, 600 and 700 L were processed in the laboratory. These samples were diluted with more water to reduce the viscosity and all samples were filtered by vacuum, precipitated with ethanol, dried and weighed. At pilot plant level, each experimental dilution was filtered in the rotary vacuum filter, using the same conditions described above. The filtration flow rate was measured for the dilutions (process viscosity indicated in brackets): 700 L (20 mPa s), 600 L (30 mPa s), 550 L (45 mPa s) and 500 L (60 mPa s). The filtration flow rate was measured every 7 min by receiving the solution in a graduated bucket during one minute. The total filtrate was also recovered and total volume was recorded.

Effect of the type of filter aid on the filtration rate

The alginate paste was diluted with water to 40 mPa s and filtered in the rotary vacuum filter, using drum rotation of 2 rpm, knife advance of 0.1 mm every 3.5 s and 14 mm thickness of filter aid. Three different kinds of filter aid were used: Celite Hyflo Super-Cel (diatomaceous earth), Celite 545 (diatomaceous earth), and Perlite (expanded lava). In each case the filtration flow rate was measured during one minute every eight minutes. The flow rates and the total filtration time were compared for each filter aid.

Comparison of filtration vs centrifugation vs centrifugation followed by filtration, with and without flocculant

In the following systems, where applicable, the filtration flow rate was measured for one minute, every three minutes during the separation of 400 L alginate extract diluted to a viscosity of 40 mPa s.

(i) Filtration

The alginate solution was filtered in the rotary vacuum filter previously covered with 14 mm layer of filter aid (Hyflo Super-Cel), with drum rotation of 2 rpm and knife advance of 0.1 mm every 3.5 s.

(ii) Filtration with flocculant

The alginate extract was filtered as above but at the same time 4 L of flocculant solution (Magnafloc 292, 0.05%) were added in the filter pan (capacity, 60 L) at a rate of 333 mL every 3 min.

(iii) Centrifugation

The alginate solution was centrifuged with an Alfa-Sharples TS-02, horizontal D-Canter continuous centrifuge, running at its maximum speed of 4200×14339, with the conveyor screw at 4180 rpm. The flow rate was regulated by a valve (9.2 to 11.5 L min^{-1}). This combination gave the best separation of solids. The quantity of solids in the centrifugate was measured every 5 min by spinning a sample in a laboratory centrifuge (2500 rpm) until the supernatant was clear. These results were expressed as the percent (V/V) of residual solids in a graduated tube. After centrifugation was complete all the separated solids were weighed; a sample was dried and weighed so the percentage (W/W) of separated solids could be calculated.

(iv) Centrifugation with flocculant

The alginate solution was centrifuged as above, but this time 4 L of flocculant solution (Magnafloc 292, 0.05%) were added into the dilution tank prior to centrifugation.

(v) Centrifugation and filtration

The alginate extract was first centrifuged, as previously described, and then the clarified extract, which contained suspended solids, was filtered as in the previous experiment.

All the experiments were run at pilot plant level in triplicate. The average, standard error and confidence
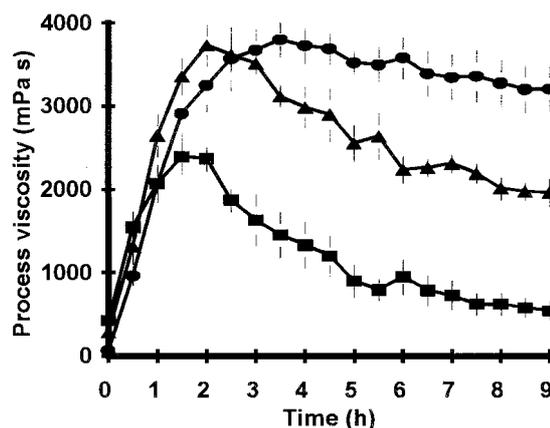

Figure 1. Effect of time on the 'process viscosity' of the alginate solution during extraction step at 70 °C (●), 80 °C (▲) and 90 °C (■). Mean ±1 SE.

interval (±1 SE) with a level of significance at 95% were computed. ANOVA was used to detect significant differences among treatments ($p < 0.05$) and Tukey honest significant test was used for post-hoc comparison of means (Zar, 1984; StatSoft, 1995).

Results

Effect of temperature and time during alkaline extraction

The maximum process viscosity during the extraction step was obtained using 70 °C with 3800 mPa s at 3.5 h. After that time, the viscosity began to decrease slowly, almost maintaining an asymptotic curve. With the extraction at 80 °C the maximum viscosity was at 2 h with 3730 mPa s, but in this case the viscosity decreased faster down to 1963 mPa s in 9 h. The fastest reaction occurred at 90 °C, giving the maximum viscosity at 1.5 h with 2393 mPa s, however this maximum process viscosity was significantly lower than in other treatments, and the viscosity decreased to 546 mPa s after 9 h (Figure 1). The filtrate viscosity varied in a similar way to the process viscosity. Using 70 °C, the maximum viscosity was 13.07 mPa s after 3.5 h; with 80 °C the maximum was 11.27 after 2 h and with 90 °C it was 8.7 after one hour (Figure 2a). Significant differences were found among all treatments.

The alginate yield increased as the process temperature was increased. In all treatments the maximum yield was obtained at about 3.5 h, after this time
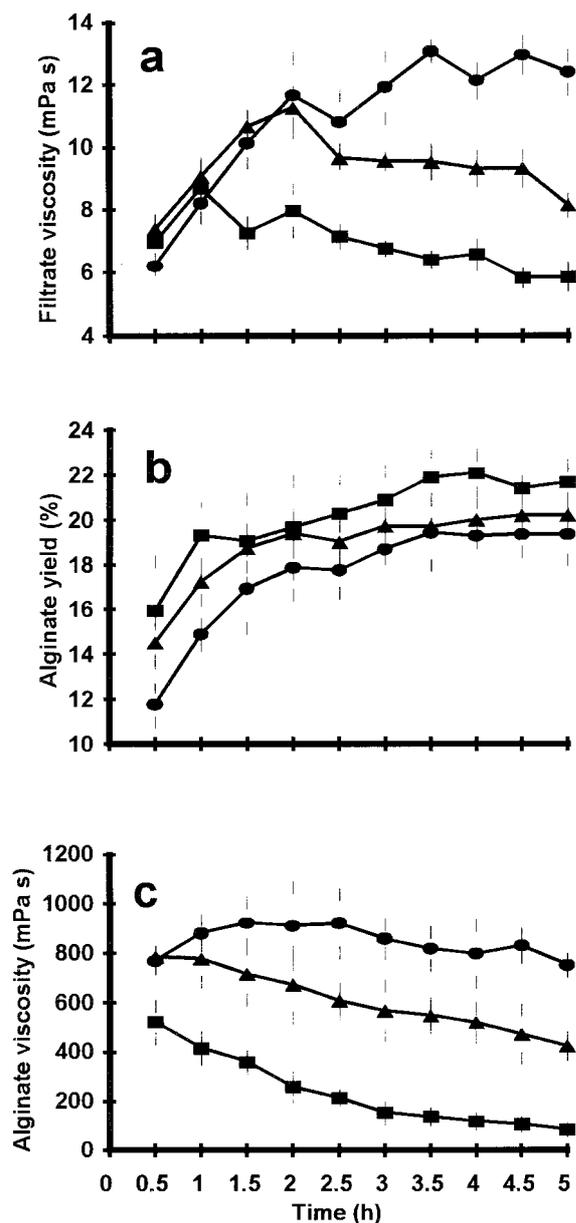
Figure 2. Effect of time during extraction step at 70 °C (●), 80 °C (▲) and 90 °C (■) on a) filtrate viscosity of the alginate solution, b) alginate yield and c) alginate viscosity (1% solution). Mean ±1 SE.

Figure 3. Effect of time on a) 'filtrate viscosity' during filtration at 70 °C. b) alginate yield (●), and viscosity of alginate (▲) precipitated from the filtrate. Mean ±1 SE.

the yields obtained did not increase significantly. The highest value obtained, 21.9%, was at 90 °C; 80 °C yielded 20.2% and the lowest yield, 19.4%,was obtained at 70 °C (Figure 2b). The yield at 90 °C was significantly higher than the yield at 70 °C.

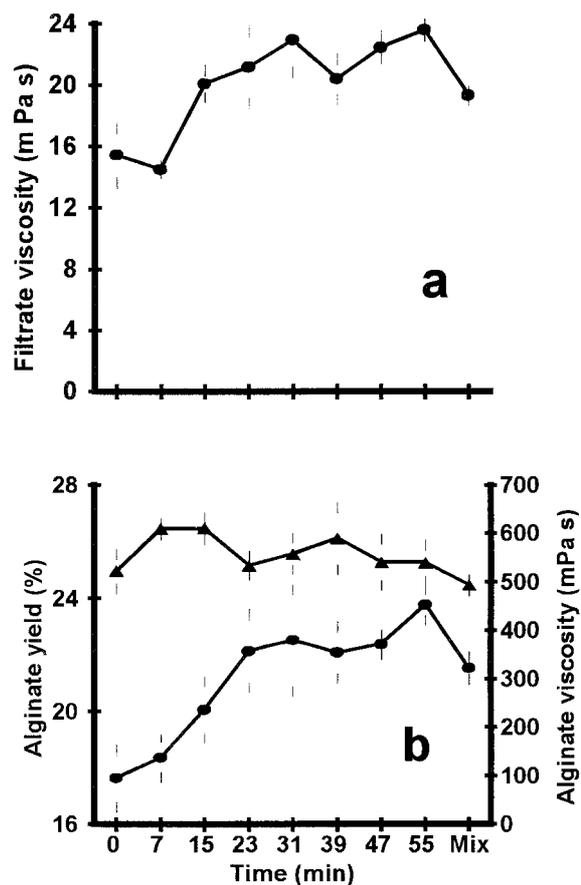The viscosity of the 1% alginate solution, prepared from the solid precipitated from the extraction filtrate, showed a constant reduction as a function of time in all treatments. This reduction was more pronounced as the temperature of the extraction treatment was increased. With the treatment at 70 °C the viscosity was almost constant, varying from 923 to 753 mPa s after 5 h of treatment. Using 80 °C, the viscosity reduction followed almost a straight line with negative slope, losing about 100 mPa s per hour; in this case the variation was from 785 to 426 mPa s. With the treatment at 90 °C the reduction was more pronounced; the alginate viscosity began at 523 mPa s and was reduced to 86 mPa s after 5 hours treatment. At 90 °C, the viscosity loss was 154 mPa s per hour, during the first two hours of treatment (Figure 2c). Significant difference was obtained among treatments.
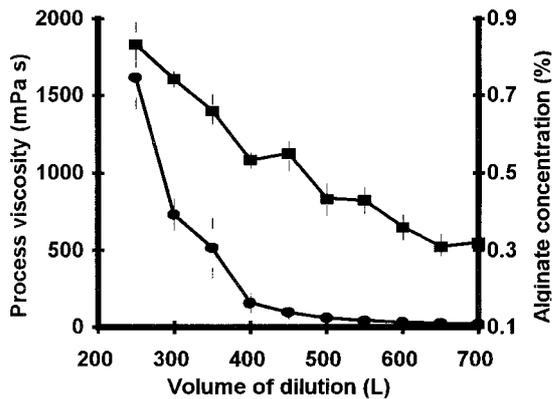
Figure 4. Effect of dilution on 'process viscosity' (●) and alginate concentration (▲) diluting to different viscosities. Measurements at 70 °C. Mean ±1 SE.
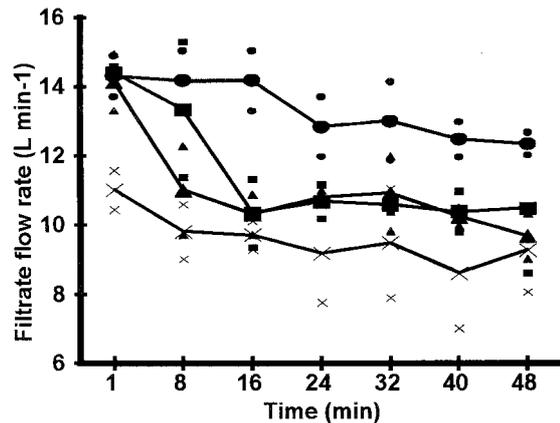

Figure 5. Filtration flow rate of the alginate extract, diluting to 20 mPa s (●), 30 mPa s (■), 45 mPa s (▲) and 60 mPa s (**X**). Measurements at 70 °C. Mean ±1 SE.

Effect of filtration time on the filtrate viscosity and alginate yield and viscosity

The diluted alkaline extract, with the residual algae still in it, had an initial viscosity of 36.6 mPa s before filtration started. The viscosity of the filtrate samples taken every 8 minutes during filtration ranged from 14.5 to 23.6 mPa s, with no significant difference from the starting point (Figure 3a). The alginate yield obtained from the filtrate started with 17.6%, and during the filtration the alginate yield increased significantly to 23.7% after 55 min, which was the time necessary to filter the 600 L (Figure 3b). The yield from the total mixed filtrate was 21.5%. The viscosity of the alginate, extracted from the filtrate samples, remained almost constant during the entire filtration time, ranging from 522 to 610 mPa s (Figure 3b).

Effect of alkaline extract dilution on the filtration rate

After alkaline extraction the viscosity in the resulting paste was 1617 mPa s. The viscosity was reduced as the amount of water was increased from 733.3 mPa s for dilution to 300 L down to 21.2 mPa s diluting to 700 L (Figure 4). The alginate concentration in solution was also reduced as dilution was increased, from 0.83% in the initial paste to 0.32% when diluting to 700 L (Figure 4). As expected, the filtration rate was higher when the solution was more dilute (20 mPa s) than less dilute (60 mPa s). Average filtration flow rate during the first 48 min ranged from 13.3 L min^{-1} (diluting to 700 L, 20 mPa s) to 9.6 L min^{-1} (diluting to 500 L, 60 mPa s) (Figure 5). Total filtration time ranged from 55 min (diluting to 20 mPa s) to 88 min
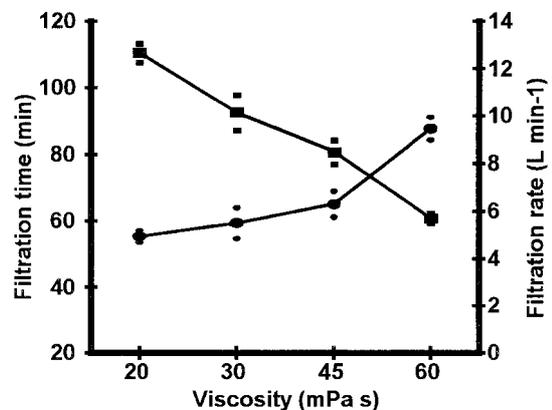

Figure 6. Total filtration time (●) and filtration flow rate based on total filtration time (■) at different viscosities. Measurements at 70 °C. Mean ±1 SE.

(diluting to 60 mPa s) (Figure 6). The filtration flow rate, calculated using the total time taken to filter the entire diluted extract, ranged from 5.7 L min^{-1} (at 20 mPa s) to 12.7 L min^{-1} (at 60 mPa s) (Figure 6).

Effect of the type of filter aid on filtration rate

The averages of the filtration rates shown in Figure 7 were 10.5 L min^{-1} with diatomaceous earth (Hyflo Super-Cel); 10.3 L min^{-1} with diatomaceous earth (Celite 545) and 9.9 L min^{-1} with expanded lava (Perlite). No significant difference was found among the different filter aids used. To filter 400 L in a filtration area of 1.15 m^2 either 5.5 kg of diatomaceous earth or 2.3 kg of expanded lava was used.
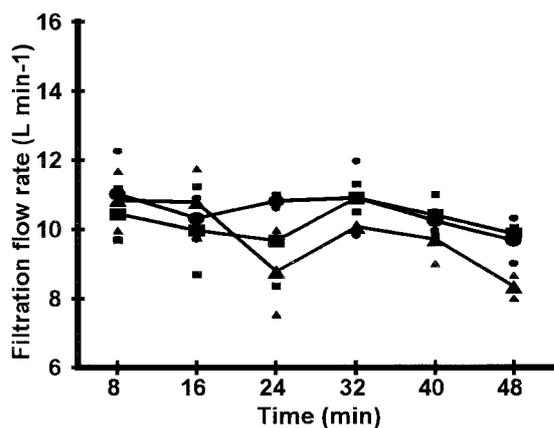
Figure 7. Effect of the type of filter aid material on the filtration flow rate. Celite Hyflo Super-Cel (diatomaceous earth) (●), Celite 545 (diatomaceous earth) (■), and Perlite (expanded lava) (▲). Measurements at 70 °C. Mean ±1 SE.
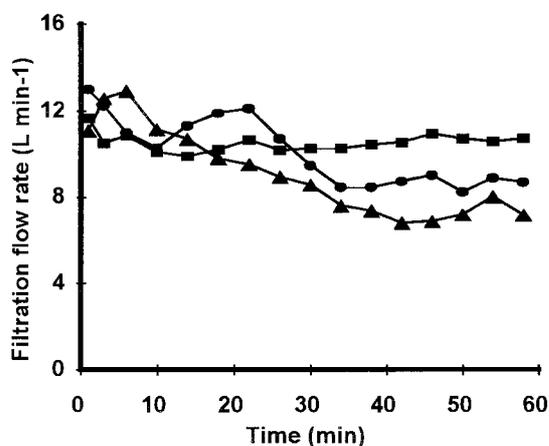

Figure 8. Effect of the separation system on the filtration flow rate. Filtration (●), Filtration with flocculant (■), Centrifugation-Filtration (▲).

Comparison of filtration vs centrifugation vs centrifugation followed by filtration, with and without flocculant

When the filtration flow rates are compared for filtration, filtration after treatment with flocculant and filtration after treatment by centrifuge (Figure 8), no significant differences were found. Treatment with flocculant before filtration did result in the removal of a drier filter cake, with less loss of alginate extract, but the quantity of diatomaceous earth used (14 mm original thickness, 3-4 mm remain on filter drum) was the same with or without treatment with flocculant. The quantity of solids separated by centrifuge was the same whether or not flocculant was added. Centrifuga-

tion at laboratory level of the liquor discharged from the industrial continuous centrifuge showed it contained 18% V/V (±1) of solids. Calculation showed that the industrial centrifuge separates 23% W/W (±2) of the total insoluble solids in the diluted alginate extract. The resulting alginate extract has poor clarity and for food grades, a filtration was needed to separate the residual fine solids. However treatment of the alginate extract with flocculant before centrifugation did allow a more constant flow rate to be maintained through the centrifuge, without the danger of the centrifugate overflowing into the solids being discharged by the screw.

Discussion

Large number of papers and patents have been published about alginate production, however few details of the reactions involved in the process have been revealed. Experimentation carried out at pilot plant level is important because the results are closer to those that can be expected at an industrial level. In the case of the alkaline extraction step, it is well known that some alkali may be used, but the proposed concentrations and times are very variable and no one author gives details of how the reaction proceeds or how the yield or quality of alginate can be affected by these parameters (Green, 1936; Le Gloahec & Herter, 1938; Bescond, 1948; Haug, 1964; Lukachyov & Pochkalov, 1965; Baranov et al., 1967; Secconi, 1967; Zvered et al., 1969; McHugh, 1987).

In the study of the effect of temperature and time during alkaline extraction, it was found that process viscosity is a good parameter to measure the progress of the reaction; this makes it unnecessary either to filter the residual algae or to isolate the alginate in order to follow the changes during reaction. In spite of having the residual algae particles present in the resulting paste, the high viscosity obtained allowed the insoluble particles to remain suspended in the liquid and the viscometer readings on any one sample remained constant for a long time. Also the results can be reproduced with very low variance. This finding is important because the method can be used to test the algae rapidly and find the best reaction time for any algal species, or for the same algae harvested during different seasons, since the alginate content may change with time (Hernández-Carmona, 1985; Rodríguez-Montesinos & Hernández-Carmona, 1991). The filtrate viscosity can also be used to fol-

low the reaction rate; however both methods reveal the same information and it is not worth spending time to filter the solution which is always a tedious step, even at laboratory level.

The results in this experiment showed that best yield was obtained using 90 °C; however the viscosity obtained after 2 hours treatment (261 mPa s) was considered too low, because a further loss of viscosity could be expected in the next steps of the process, particularly during the conversion from calcium alginate to alginic acid. Using 80 °, the yield obtained was high enough after 2 h and the viscosity was 673 mPa s, leaving a reasonable margin for further viscosity loss, but still having a useful product. The treatment at 70 °C required 3 h extraction to obtain a yield similar to the treatment at 80 °C for 2 h. The viscosity after 3 hours at 70 °C was higher than 2 h at 80 °C but in a commercial process the extra cost of heating for another hour is not worth the difference in viscosity, unless the objective is to produce a very high viscosity product.

Haug (1955) noted that for *Laminaria digitata*, the temperature of extraction did not influence the viscosity of the alginate obtained, when kept below 50 °C, but when the temperature was as high as 75 °C the viscosity of the extracted alginate decreased markedly. However our results demonstrate that alginate from *Macrocystis pyrifera* can resist temperatures up to 80 °C without marked reduction in viscosity if the extraction time is no greater than two hours. The high yield obtained using one acid washing at pH 4 during pre-extraction and extraction in hot conditions (as described in this paper), confirms our previous finding that alginate can be extracted with good yield (20.2%), with only a minimum amount of calcium ions exchanged during the acid pre-extraction treatment (Hernández-Carmona et al., 1999).

The control of the alginate viscosity is a very important factor for the industry, because the use of the alginate depends mainly on its viscosity, (although the calcium content and mesh size are also considered). Generally companies offer three main categories of alginate (though many other categories can be found in between): alginates with high viscosity (above 800 mPa s), medium viscosity (400–800 mPa s) and low viscosity (below 400 mPa s) (Protan, 1983; Kelco, 1984; Kimitsu, 1998). The following are only some examples of the many applications of alginates: high viscosity alginate is used in food, cosmetics and dental impression compounds; medium viscosity alginate is used in pharmaceuticals, substitutes for blood plasma and pet food; low viscosity alginate is used in paper

making, fruit industry and textile printing and dyeing (Protan, 1983; Kimitsu, 1998). The curves obtained in these studies of the extraction process (Figures 1 & 2) provide useful information for the control of the viscosity of the final product. Varying the time and temperature of this step can very closely predetermine variations in yield and viscosity of the resulting alginate. However the best compromise between yield and viscosity is to carry out the alginate extraction at 80 °C for two hours at pH 10.

The next experiment investigated whether the extracted alginate could be degraded during the time that the diluted extract was filtered. The results show that, by maintaining the temperature at 70 °C, no significant differences in viscosity were obtained between the samples taken as filtration progressed, indicating that no significant degradation of the product occurred. The viscosity of the total mixed filtrate coincided with the average of the seven different filtrate samples taken during filtration. The alginate yield obtained from the filtered sampled was 17.6% at zero time, which corresponds approximately to the value obtained in the extraction experiment after 2 h at 70 °C. During the filtration, the alginate yield increased from 17 to 23.7% after 55 min, which was the filtration time for the 600 L. The yield in the final mixed sample of filtrate was 21.5%, coinciding with the average of the different samples taken during filtration. The increase in yield indicates that alginate was diffusing into the solution during the residence time in the dilution tank, because of the agitation effect, and therefore the filtration step not only is useful in removing the residual algae but also allows an increase in the alginate yield. The viscosity of a 1% alginate solution, using alginate isolated from each filtrate sample, remained almost constant throughout filtration, ranging from 522 to 610 mPa s; therefore no significant degradation occurred on the alginate during this step. This result confirms the finding from the extraction experiment, that alginate is very stable at 70 °C. It is concluded that the filtration step not only allows the separation of the insoluble residues of algae, but also allows the alginate to diffuse into the solution, increasing the yield with no significant degradation of the product.

The alkaline extract usually has a paste consistency and must be diluted before the insoluble algal material can be separated from it. Water consumption is always an important consideration in alginate production, often it must be minimised. Also the larger the volume of diluted extract, the longer it may take to process through the separation equipment. Therefore the aim

of the next experiment was to find the minimum dilution that would allow satisfactory separation of the algal residue. The studies were made using the rotary vacuum filter and the best compromise between flow rate and water usage was found to be a dilution of the alkaline extract to a viscosity of 45 mPa s. With a viscosity of 60 mPa s the pipe that fed the pump to the filter sometimes blocked. Dilution to viscosities of either 30 or 45 mPa s gave similar flow rates (Figure 5) and, while a dilution to 20 mPa s gave even faster flow rates, the larger volumes caused capacity problems with pumps and tanks of the pilot plant. Other factors that may affect the filtration rate on the rotary vacuum filter are the filter aid used, the knife advance speed and drum rotation speed. The knife advance must be keep as slow as possible to save filter aid, yet still keep a clean surface available. The knife advance programmed to a speed of 3.5 sec per mm gave good results. The drum rotation must be adjusted by visual determination, as fast as possible, but ensuring that the shaved cake keeps partially dry and can be removed uniformly. For dilutions to 30–45 mPa s, the best drum rotation was 2 rpm, but for dilution to 60 mPa s, the rotation was reduced to 1 rpm in order to get the same result.

No significant differences in flow rates were found between the three filter aids used. Therefore any decision taken would depend on the amount and cost of the material used. Based on the yield of alginate obtained (21.5%), to produce one kilogram of alginate, 2.5 kg of diatomaceous earth (Celite 545) was needed, at a cost US $1.25 per kilogram of alginate produced. For expanded lava (Perlite) 1.0 kg per kilogram of alginate produced was used, costing US $1. Therefore the best decision is to use Perlite as a filter aid.

Clarification of the alginate extract is a very important factor to consider during alginate production. These results show that filtration is the best method to clarify the diluted extract. However some considerations must be noted. The use of flocculant during filtration does not improve flow rate but it does improve the shaving of the filter cake, yielding a drier, more uniform sheet and therefore resulting in less loss of alginate in the residues. While flocculant does not improve the separation of solids in centrifugation, it does allow a more constant flow through the centrifuge, reducing the danger of flooding the bowl and losing alginate extract with the solids being discharged by the conveyor screw. A high percentage of fine insoluble solids remain in the centrifugate so this system can only be used for industrial grade alginate, with

uses where the presence of solids do not interfere in the application (e.g. pet food).

The combination of centrifugation followed by filtration offers no advantages because filtration time and flow rate were about the same as for filtration alone, and the same quantity of filter aid was required. The only advantage is that centrifugation can remove any coarse solids that might otherwise block the pump. In spite of the negative results obtained with the use of centrifugation and flocculation/centrifugation, these results are useful in that they warn the industry against the use of these systems, unless the objective is the production of technical grade alginate that could be cheaper because of the lack of use of filter aid, since the latter represents a significant cost in alginate production. When the filtration flow rates in Figure 8 are compared with those in Figures 5 and 6, it is apparent that filtration flow rate depends more on the viscosity of the alkaline extract being filtered rather than the quantity of solids in the extract, at least for the level and type of solids encountered here.

Acknowledgements

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