Bull. Fish. Res. Stn., Sri Lanka (Ceylon), Vol. 23, *Nos.* 1 *&* 2, *pp* 29-35, *June & Dec.*, 1972

Agar from *Gracilaria Lichenoides* and *Gracilaria Confervoides* from Ceylon

By

M. Durairatnam[1], J. Grero[1] and P. Wimalasiri[2]

INTRODUCTION

" Agar-agar " is a name derived from the jelly obtained from certain red algae in Malaya. Agar was produced in India from *Gracilaria confervoides* Rao, Patel, and Shah 1965 and Umamaheswara Rao 1969, 1970. In Ceylon agar is prepared from the red seaweeds *Gracilaria confervoides* and *Gracilaria lichenoides*. They are commonly known as Ceylon moss and the local names are " kandha parsi or sanchow parsi ". Before the war agar was mainly imported from Japan. During the war small quantities of *Gracilaria lichenoides* collected from a small island called Pallaithivu about seven miles from Jaffna was exported to India. In 1952 the Ministry of Industries and Fisheries became interested in the study of marine algae of economic importance. Accordingly a systematic survey was carried out in the first instance for seaweeds producing agar. Durairatnam and Medcof, 1954, reported on the Ceylon's Red Seaweed Resources which was confined to agar producing seaweeds. It was found that *Gracilaria confervoides* was found in large quantities at Koddyar Bay, Trincomalee, while the other species was found in Puttalam lagoon, Mannar, and some islands off Jaffna. Durairatnam 1954, 1955, determined the gel centent and the cost of collection, production and bleaching of *Gracilaria confervoides* and found that the weeds were of sufficient quality to be exported. Durairatnam 1961, after a systematic survey of the marine algal resources of Ceylon reported on several species of agar producing seaweeds specially species of *Gracilaria*, *Hypnea* and *Gelidiella* but since only *Gracilaria confervoides* and *Gracilaria lichenoides* were found in large quantities and contained high gel content, they were investigated in detail. Durairatnam 1965, went into the question of conservation of *Gracilaria confervoides* at Koddyar Bay and studied the best time for harvesting the seaweeds. A small scale manufacture of crude agar from *Gracilaria* seaweeds was carried out by Gunasekera 1963.

Chemical analysis of *Gracilaria confervoides* showed it to be a rich source of agar and an export market was found for this commodity in Japan, but due to unscruplous traders exporting seaweeds mixed with large quantity of impurities the export trade came to a halt.

Efforts are now being made to produce agar in Ceylon to meet the local demands and to export any surplus if available. This is a joint venture by the Industrial Development Board and the Department of Fisheries.

Uses of Agar

1. *In brewery.*—agar solution is used to coagulate suspended impurities and make them precipitate.

2. *In the manufacture of jam.*—Agar is used as a viscosity supplement, especially in fruits where pectin does not show sufficient viscous property.

---

1 Fisheries Research Station, P.O. Box 531. Colombo Sri Lanka (Ceylon.)

2 Industrial Development Board 16, Gregory's Road, Colombo 7, Sri Lanka (Ceylon.)

3. *Anticlotting agent of blood.*—Sulphuric ester of agar is used in medical operation since it shows anticlotting property of blood.

4. *Printing.*—Agar-agar is used as a printing block. Agar gel with sugar or gelatine is poured into a flat plate and allowed to coagulate. The paper on which articles are written with special ink is placed on the gel. The ink is absorbed by the agar gel which is used as a printing block.

5. *Meat preserving.*—Agar is used in canning of meats to give it a good appearance and to remove excess of moisture from the meat while retaining it in the can.

6. *Spices and condiments.*—Agar is used as a stabiliser in mayonnaise and in vegetable pastes where its water absorbtive action is also necessary.

7. *Bacteriological media.*—Agar is used as a medium for the culture of bacteria for medical and brewery investigations. Vaccines such as cholora, gonococcal, typhoid, paratyphoid A and B are prepared by growing cultures of the required organisms on agar surfaces with suitable medium.

8. *Dental impression materials.*—There are several patents for dental impression materials using agar as a base.

9. *Medical research.*—Agar is used as a medicine for loose bowels, toxication caused by eating fish. Although agar is not considered to be nutritious it coagulates all toxins in the organs of the body to be excreted. It is used by fat women to become slim. Dry agar is also used as a plaster for wounds since it does not rot. It is used in surgical dressings because of its anti-coagulent properties. Many laxatives consist of liquid paraffin as a base with agar.

10. *Confectionary.*—There are a large number of confectionaries where agar-agar is used, since it does not hydrolise with age as in the case of gelatine. It is used as a stabiliser in ice creams and is used in aerated waters and fruit juice, cordials and jelly food packs. It is used in fish preservation, vegetable pastes, vinegar and wine manufacture, deserts and salads.

11. *Other uses.*—Agar is used in leather and cotton dressings, photographic films, chemical and physical uses, e.g., pH determination, studies in diffusion, electrochemistry and research on the properties of gels and sols. It is used in the manufacture of tooth paste. In textiles it is used as a thickener in dyeing and printing. It is used in the sizing of paper as it prevents the formation of wax or grease. It is also a component in soaps, creams, cosmetics, shoe polish and hand lotions. It is used in the manufacture of plywood and as a constitutent of high grade adhesives. Agar gel serves as a medium in the use of graphite as a lubricant in the hot drawing of tungsten wire for electric lamps.

MANUFACTURING PROCESS IN CEYLON

This work was carried out in the laboratory of the Fisheries Research Station periodically. The seaweeds used for this purpose was *Gracilaria confervoides* collected at Koddyar Bay, Trincomalee.

Treatment of Seaweeds prior to extraction

The seaweeds collected were washed for periods ranging from 3 hours to 24 hours. It was was observed that the yield of agar as well as the gel strength decreased with increase in time of washing as shown in Table I. This probably due to the water soluble fraction being removed.

TABLE I

Showing % yield of agar and agar strength in grams after different periods of washing

Duration of washing	*Crude agar* —	*Fine Agar* —	*Gel strength of fine agar* %
1 Hour ..	54·1 ..	31·2 ..	4013
3 Hours ..	49·6 ..	28·0 ..	36·5
6 Hours ..	26·4 ..	22·1 ..	23·4
12 Hours ..	22·7 ..	20·3 ..	17·5

The results are the average of a number of experiments.

From the above experiments it is suggested that to get a sufficiently pure product the weeds should be washed for a period of three hours in six tubs the last two being lined with marbled slabs. The period of washing should be thirty minutes in each of the first five tubs, and then allowing it to remain in the last tub for thirty minutes, as this will result in the production of a slightly clear colourless product. After washing the seaweeds they are dried in the sun for about three days. The dried bleached seaweeds are stored in a ventilated room and should be utilised for processing as soon as possible. If it is not intended to use the weeds immediately the weed should not be completely washed as to remove too much salt as the salt keeps the weeds moist during storage and prevents acid fermentation. The unbleached seaweeds can be kept without deterioration for about two years if stored in a dry place.

Experiments were conducted on a commercial scale at Mutwal factory and as reported by Wood 1946 the weeds acted as an insulating material and cold spots developed in the boiling vats. A series of experiments were carried out in extracting gel from unminced weeds and minced weeds. It was found that the maximum yield of agar with unminced weeds was 31.2% and minced weeds 23.4%. As such it would be advisable to use the weed as it is, without mincing. The cold spots in the boiling vats could be avoided by stirring the weeds vigorously.

2· Extraction of Agar from Gracilaria confervoides

(*a*) 4% of the weeds in water were extracted for 3 hours at pH. 6.5 which were filtered, frozen thawed and dried. Extraction temperatures were 95°C, 100°C, 105°C and 110°C. The results are shown in Table 2.

TABLE 2

Tests of Effects of Pressure and Temperature of Boiling

Pressure	*Temperature*	*Extraction* —	*Gel strength* —	*Viscocity*
Atmospheric ..	95°C ..	32·2 ..	38·9 ..	1·5
Atmospheric ..	100°C ..	31·1 ..	35·6 ..	1·5
2½ lbs. ..	105°C ..	23·7 ..	34·3 ..	1·6
5 lbs. ..	110°C ..	24·3 ..	17·8 ..	1·8

It will be seen that the percentage of agar extracted was greater at atmospheric pressure and at a temperature of 95°C and with increase of temperature and pressure the percentage of agar decreased. As such it would be advisable to use open vats lined with stainless steel.

(*b*) *Time of extraction.*—Several experiments were conducted to find out the time of extraction to obtain the maximum yield of agar and the results are given in Table 3.

TABLE 3

Results of Tests to determine suitable extraction time of Seaweeds using a 4% mixture of weeds in water

Experiment	*Time in Hours*	*Agar* —	*Gel strength* —	*Relative viscocity*
1	1	25.8	36.5	1.5
	2	28.3	34.1	1.5
	3	31.2	40.2	1.7
	4	298.7	33.5	1.6
2	1	27.5	33.8	1.5
	2	28.1	34.2	1.6
	3	29.8	36.5	1.6
	4	27.2	30.8	1.4
3	1	26.3	37.2	1.4
	2	28.1	37.0	1.5
	3	30.8	38.3	1.6
	4	27.1	36.9	1.4
4	1	25.8	37.8	1.5
	2	28.3	38.6	1.6
	3	31.1	44.2	1.7
	4	27.9	37.4	1.5

It will be observed from experiments 1 to 4 that the most suitable time for boiling the weeds is three hours as it gives the maximum yield of agar. According to Gunasekera 1963, a 4% (4 lbs. per 10 gallons) mixture of weeds in water had to be extracted twice to recover all the agar, showing that 2 more dilute mixture 2% (2 lbs. per 10 gallons) in 4 hours could effect complete extraction and the liquid set easily into a firm jelly, a form required to effect purification. When a 4% mixture of weeds in water is used a second extraction should be done using the residue left from the first extraction for a period of 1 or 2 hours.

However a 4% mixture of weeds in water is recommended since the thawed gel will not be continuous when the mixture of weeds in water is below 4%.

(*c*) *pH extraction.* Filteration was easiest when the pH of extraction of the weeds is maintained between 6 to 6.5 especially when acid Sodium phosphate was used (1 part in 1,200).

Extracting vat

The vat should be a double jacketed vessel lined with stainless steel and a boiler 15 P. S. I. generating about 70 to 100 lbs. of steam per hour may be used for heating the vessel.

The extracting temperature should be in the range of 95°C to 100°C. Press extractor is unsuitable for *Gracilaria* since the outer skin is soft as compared to *Gelidum*. If pressure is used agar molecule will be destroyed and the quality of production will be reduced as such an open type extractor is recommended. During filteration the temperature should be kept as high as possible and the pressure as low as possible since the setting temperature and viscosity increases with pressure. *Gracilaria* agar sets between 45°C-50°C. The primary filteration is performed by installing a suitable strainer in the extracting vat so that the residue may be removed and boiled again. The secondary filteration is carried out through fine cloth filters using filter aids and activated carbon for clarification. The filtered liquid is collected in aluminium trays and allowed to remain overnight where it sets into a firm gel.

Dehydration. The filtered liquid contains water and impurities which reduce the gelling power. The best method of dehydration and removal of impurities is the refrigeration method. The trays containing the firm gelly may be placed in cold rooms or large refrigerators where the temperature is up to –10°C. for a period of 1–3 days. It was found that agar gelly containing 98% moisture was reduced to 36%, Gunasekera 1963, showed that using an icemaking machine it was found that 3 days freezing was necessary to freeze a 56 lb. block of gelly before it was possible to separate ice and agar within the block of gelly.

Drying

There are several methods of drying as reported by Wood 1945.

(*a*) Spray drying ;
(*b*) drum drying ;
(*c*) tunnel drying ;
(*d*) drying infra red light.

It is suggested that the infra red light method be adopted in Ceylon but the temperature should not exceed 50°C. using infra-red lamps 1,200 watts. Agar which is one inch thick could be dried in four hours. By using infra red lamps and a moving belt it would be possible to adjust the lamps close to the wet part which is far away from the dry end of the belt. The agar gel should be dried on cheese cloth. To ensure rapid drying of agar the thawed agar should be dipped in alcohol before drying. This also removes much of the colour in the finished product.

In India the thawed agar is dried on cheese cloth in sun light. This could be adopted in Ceylon but drying takes a long time about three days or more.

The dried agar is crushed into powder and packed in polythene bags.

SUGGESTED METHODS OF MANUFACTURE OF AGAR IN CEYLON

The dried unbleached seaweeds received in the factory should be washed in small quantities at a time preferably 50 lbs. The washing should be done in 6 stages in tubs with outlet pipes. The last two tubs should be lined with marble slabs. The weeds should be thoroughly washed for 30 minutes in each tub and allowed to remain in the last tub for 30 minutes preferably in rain water. It should then be dried for about 3 days in bright sunlight. 27·27 Kg. (60 lbs.) of washed and dried seaweeds is placed in a double jacketed open pan extractor lined with stainless steel with 675 litres of water (150 gallons) and heated by steam from an oil fired boiler 15 P. S. I. generating about 70 to 100 lbs. of steam per hour. The pH is maintained at about 6 to 6·5 by adding acid Sodium phosphate (1 part in 1,200). The weeds are boiled for about 3 hours. Since the concentration of weed to water is 4% the weed should be boiled twice showing that a more dilute mixture would be better. The alternative is to use a 2% concentration of weed to water, i.e., to use 13·64 Kg. (30 lbs.) of the weed using 75 liters (150 gallons) of water and boiled for 2 hours. The extraction temperature is maintained between 95–99°C. The liquid should be vigorously stirred constantly to prevent cold spots in the boiling vats. The boiled liquid is run into another steam heated filtering vat also lined with stainless steel. The filteration is done by placing cheese cloth over a sieve embedded in the vat. After adding filter aid and activated carbon, the filterate is pumped into aluminium trays and left overnight to form a firm gel. The gel is cut into strips and allowed to freeze in the cold rooms or a large refrigerator where the temperature can be adjusted up to 10°C. for about 24 hours. The frozen agar is thawed in water at room temperature. As the ice melts the dissolved impurties are removed and the pure gelly is left behind. To remove excess of water the gelly is dipped in alcohol and dried under a battery of infra red lamps or in sunlight. When the gelly is completely dry it is ground into powder and packed in cellophene bags.

METHOD OF AGAR MANUFACTURE ON A COMMERCIAL SCALE

Raw *Gracilaria confervoides*

↓

Washing in fresh water
5 washes of 30 minutes in each tub

↓

Soaking in rain water or soft water
30 minutes

↓

Drying and bleaching for 3 days in
sunlight

↓

Extraction of 60 lbs. (27·27 Kg. with 150 gallons) (675 litres) of water in double-jacketed open va
lined with stainless steel heated by steam. pH maintained at 6 to 6·5 using
acid Sodium phosphate (1 part in 1,200)

↓

Filtering
in double-jacketed open vat lined with stainless steel by placing cheese cloth with filter aid and
tivated carbon on sieve embedded in vat

↓

Cooling the filtrate in aluminium trays at room temperature
for 24 hours

↓

Freezing the agar for 24 hours in cold rooms or refrigerators

↓

Thawing in water at room temperature

↓

Dipping thawed gelly in alcohol and drying in sunlight or using
battery of infra red lamps

↓

Agar-Agar

(18 to 20 lbs. or 8·8 to 9 Kg.)

BIBLIOGRAPHY

DURAIRATNAM, M. and MEDCOF, J. C., 1954. Ceylon's Red Seaweed Resources. *Ceylon Trade Journal*, Vol. 19, No. 4, pp. 1-6.

DURAIRATNAM, M. and MEDCOF, J. C., 1955. Ceylon moss—A marine resources. *Prog. Rpt., F. R. S., Ceylon*, No. 1. pp. 13–16.

DURAIRATNAM, M., 1961. Contribution to the study of the marine algae of Ceylon. *Bulletin No.* 10, *Fisheries Research Station, Ceylon.*

DURAIRATNAM, M., 1961. Economic marine algae of Ceylon. *I. P. F. C. Occasional Paper*, 61/4, 6 pp.

DURAIRATNAM, M., 1965. The Ecology of *Gracilaria verrucosa* (Hudson) Papenfuss, in Koddiyar Bay, Trincomalee. *Bull. Fish. Res. Stn., Ceylon*, Vol. 18, No. 1.

GUNASEKERA, C., 1963. Small scale manufacture of crude agar from *Gracilaria* seaweeds. *Bull. Fish. Stn. Res., Ceylon* Vol. 16, No. 1, pp. 49–52.

RAO A. V. PATEL, K. N. and SHAH, H. N., 1965. Manufacture of Agar from Red Seaweeds. *Research and Industry*, Vol. 10, No. 5, pp. 131–133.

PILLAI, V. K., 1955. Water soluble constituents of *Gracilaria lichenoides. Journ. Sci. Indust. Res.*, 14 B, 473–477.

UMAMAHESWARA RAO, M., 1969. Agar and algin producing seaweeds of India. *Proc. 6th International Seaweed Symposium*, pp. 715–721.

UMAMAHESWARA RAO, M., 1970. Economic Seaweeds of India. *Bull.* 20, *Central Marine Fisheries Research Institute, India.*

WOOD, E. J. F., 1946. Agar in Australia. *C. S. I. R. (Aust.) Bull.*, 203, 43 pp.