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Upgrading Solar Salt by Mechanical Washing

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ABSTRACT

Salt is exclusively manufactured in India by solar evaporation of seawater and/or inland brines. The solar salt thus produced is 96–98% pure and is associated with 2–4% of impurities. These impurities are salts of calcium and magnesium, present as sulphates and chloride along with some insoluble matter. Soda ash and caustic soda manufacturers in India, consume about 330,000 t of solar salt. Due to imposition of strict pollution control in the last 5–7 years, these industries demand a having quality conforming to BIS:1982 which can reduce the chemical treatment cost and produce less waste material such as precipitates of magnesium hydroxide, calcium carbonate and barium carbonate. Mechanical washing of solar salt is a low cost process which can be adopted by many large and medium-scale salt manufacturers in India. A bench-scale unit has been designed and fabricated. Experiments conducted on this unit have shown that the freshly harvested solar salt can be upgraded to the quality required by the soda ash and caustic soda chlorine industry and to meet specifications of salt for the chemical industry specified by Bureau of Indian Standards BIS:1982 for grade II and very near to grade I. The estimated cost of washing is Rs. 55–63 per t on plant producing 5–15 t/h and savings in the cost of chemical treatment is about 40%.

INTRODUCTION

Salt, a commodity of vital importance for human consumption and a basic raw material for the alkali and other chemical industries, is manufactured exclusively from seawater and inland brines by solar evaporation in India. The important solar salt producing centres are shown in Fig. 1. Seawater is a complex aqueous system and the separation of various salts from this system is governed by phase rule, i.e. the mutual solubility relationship of various salts present in it. Based on this, several workers (Garrett, 1966) have studied the chemistry of separation of salts at various stages of evaporation. Fortunately, it is possible to control the solar salt works operation to win the common salt and other co-product evaporites based on density control, i.e. by determining the density of brines at various stages of evaporation by Baume hydrometers. Due to mutual solubility, there is always an overlap of deposition of one salt over the other. Thus the salt manufactured by solar evaporation of seawater or other brine sources is always contaminated with common impurities like calcium sulphate due to co-crystallization and magnesium sulphate and magnesium chloride due to adhering mother liquor and insoluble matter during harvesting of salt from mud pans and atmospheric dust deposition. The extent of impurities contamination depends upon the method of salt production and has been found in the range of 2–4%. The quality of solar salt produced in India is given in Table 1.

There has been a marked growth in the development of the salt industry in India after independence. Production has increased from 2.0 Mt in 1947 to 9.0 Mt in 1990 (Fig. 2). However, there are only 25 to 30 large salt producing units in the country which have an annual production of 100,000–150,000 t of salt which contributes about 40% of total production. The remaining 60% of solar salt is manufactured by some 9,075 small units. At large salt works, a comparatively good quality of salt is produced by better density control but the salt produced by smaller units is of inferior quality.

Until recently, the requirement of high purity salt for the caustic soda chlorine industry was not felt but due to the imposition of strict pollution control rules for mercury pollution and with the advent of membrane cell technology for the chlor-alkali manufacture, there is now a need for high purity solar salt. The existing solar salt works are not tuned for the manufacture of high purity salt having NaCl 99.5%

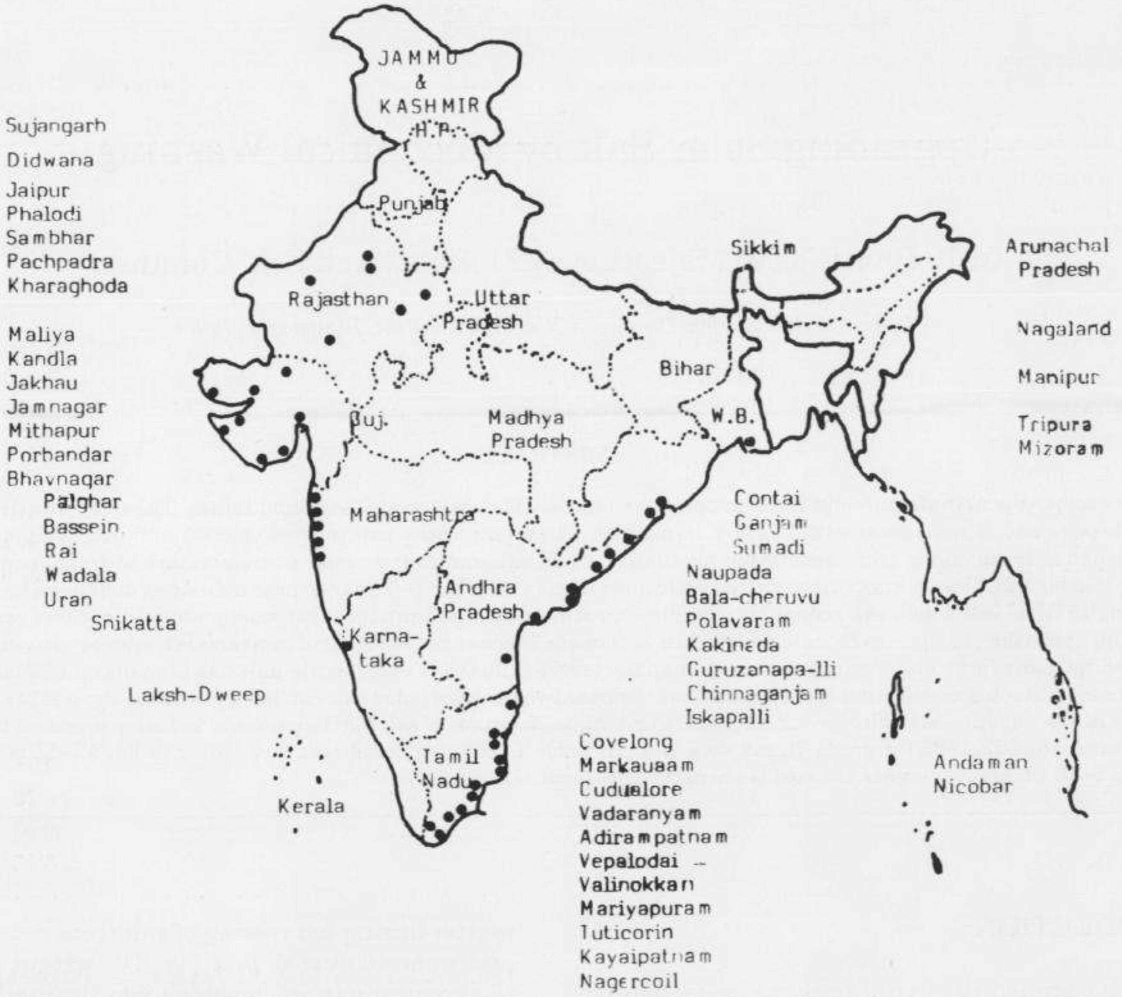

Fig. 1. Salt producing centres in India.

and above. This has been possible for developed countries like the USA (Woodhill, 1961), France (de Flers, 1966), Australia (McArthur, 1980) and Mexico (Juan, 1985) due to strict operational control during solar salt manufacturing process, followed by washing of freshly harvested salt i.e. the washing process is the integral part of solar salt manufacture in these countries. If India desires to manufacture high purity salt, it will be necessary to adopt the same measures.

EXISTING SALT UPGRADING METHODS IN INDIA

In some of the large and medium-scale solar salt works, the harvested salt is now washed by fresh saturated brine, before it is lifted from crystallisers and transported to a platform for heaping. Usually this heaped salt is then exposed to natural rains, whereby it is further upgraded. This helps the removal of magnesium impurity, but calcium and insoluble matter are slightly increased in this type of washing. One of the authors had studied the rain washing of heaped solar salt. The results are shown in Table 2.

For the rapid disposal of manufactured salt, some solar salt works carry out the artificial spray washing of small heaps at crystallisers or on platforms by fresh water or seawater. This method also helps the removal of magnesium, but calcium and insoluble matters are not removed in this type of washing.

Mechanical washing has not yet been adopted by salt manufacturers in India. Therefore, several soda ash and caustic soda companies have installed mechanical washeries at factory sites for own captive use in order to reduce chemical treatment costs.

M/s. Saurashtra Chemicals (soda ash factory) at Porbandar (Vakil, 1964) installed a screw-conveyor washery with a 600 t/day capacity. The washing was carried out by co-current methods. The salt was

TABLE 1

Quality of salt produced in India.

Sr. No.	Source	NaCl (%)	Ca^{2+} (%)	Mg^{2+} (%)	SO_4^{2-} (%)	Insoluble (%)
1.	Gujarat (seawater)					
	Bhavnagar	98.0–98.5	0.11–0.14	0.2–0.5	0.7–1.0	0.1–0.15
	Dahej	97.0–98.5	0.15–0.20	0.2–0.5	0.8–1.0	0.1–0.20
	Jamnagar	97.5–98.5	0.12–0.16	0.2–0.90	0.2–0.9	0.1–0.15
	Kandla	97.5–98.0	0.15–0.20	0.2–0.4	0.7–0.9	0.1–0.20
	Porbandar	98.0–98.5	0.13–0.17	0.2–0.4	0.7–0.9	0.1–0.20
	Dhrangadhra (inland brine)	96.5–97.5	0.2–0.4	0.25–0.5	0.9–1.75	0.1–0.20
	Kharaghoda (inland brine)	96.5–97.5	0.25–0.55	0.15–0.3	0.85–1.5	0.1–0.20
2.	Maharashtra	96.5–97.0	0.15–0.25	0.5–1.1	1.25–1.75	0.1–0.20
3.	Tamilnadu	96.0–97.0	0.15–0.30	0.25–0.6	1.0–1.5	0.3–0.70
4.	Andhra Pradesh	96.5–97.5	0.22–0.25	0.20–0.5	1.0–1.5	0.4–0.80
5.	Orissa	96.5–97.5	0.15–0.25	0.25–0.6	1.7–1.1	0.9–1.25
6.	West Bengal	97.5–98.5	0.18–0.22	0.15–0.3	0.7–0.9	0.4–0.50
7.	Rajasthan (inland brine)					
	Pachbhadra	97.5–98.5	0.15–0.25	0.2–0.4	0.7–0.9	0.1–0.20
			Na_2SO_4	Na_2CO_3		
	Didwana	96. 8	1.87	0.23	–	0.94
	Sambhar Lake	98.8	0.35	0.47	–	0.26
	Pan	98.0	0.85	0.75	–	0.30
	Resta	94.4	2.76	2.21	–	0.46

TABLE 2

Rain washing of heaped salt (experimental salt farm)

	Analysis of salt		% Removal of impurity
	Unwashed salt	Washed salt	
NaCl	97.25	98.8	–
Ca^{2+}	0.18	0.21	–
Mg^{2+}	0.35	0.06	83
SO_4^{2-}	0.75	0.60	20
Insolubles	0.09	0.13	–

washed with saturated brine in the first screw conveyor and by water in the other screw-conveyor. The wash-liquor from the washed salt was separated in the vibrating drainer and was recycled after settling part of the wash liquor and purging to maintain the magnesium concentration. This unit is no longer operating. M/s. Gujarat Heavy Chemicals a soda ash factory located at Sutrapada near Veraval in Gujarat State (Bhatt et al., 1989) has installed a mechanical washery of 2,400 t/day capacity. The technology is imported from The Netherlands. Here, the ground common salt is mixed with wash-liquor (saturated brine) from the main tank in the ratio of 1:3 to 1:4 and slurry is pumped through a long pipeline to a hydrocyclone. The hydrocyclone clarifies the salt and wash-liquor. The impurity loaded wash-liquor goes back to main tank. The salt from the hydrocyclone is led to a sieve band in slurry form where further draining takes place and then falls in a vertical column. Here it is washed with pure water in a counter-current manner. From this column, the wash liquor overflows and goes to the main tank. Part of the wash-liquor from the hydrocyclone is purged to maintain the magnesium balance in the wash-liquor. In the process, salt is not centrifuged but goes for direct dissolution from the vertical tank for processing to manufacture soda ash. It is claimed that about 40–50% of chemical treatment cost is saved by washing the salt in this unit.

M/s. Gujarat Alkalies (Dept. of Scientific & Industrial Research, 1991) and Chemicals Ltd., at Baroda (Gujarat), a public sector company manufacturing caustic soda has recently installed a mechanical washery of 40 t/h capacity. The technology has been imported from M/s. Krebs, Swiss. Here the salt

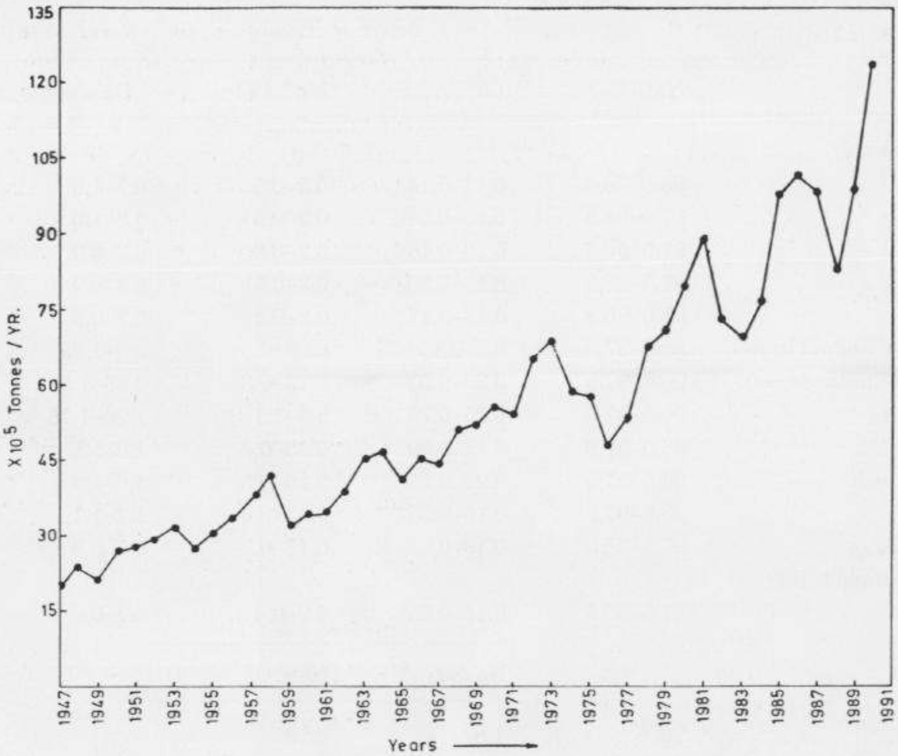

Fig. 2. Salt production in India.

TABLE 3

Salt washing 40 t/h capacity at GACL, Vadodara

	Percent by wt. dry basis	
	Raw salt	Washed salt
NaCl	97.80	99.50
$CaSO_4$	0.70	0.28
$MgSO_4$	0.29	0.03
$MgCl_2$	0.18	0.18
Insolubles	1.03	0.02

is ground and mixed with the saturated wash-liquor in an elutriator in a counter-current manner. Part of the calcium sulphate and insoluble matter is removed here. From the elutriator, the salt is led to an agitator, where it is further washed with brine obtained from the next stage. It is then placed in a hydro-extractor and finally centrifuged. The typical analysis of raw salt and washed salt are given in Table 3. The loss of salt is 10–11%. The savings in the cost of chemical treatment is 22% percent.

CSMCRI, Bhavnagar has carried out extensive R&D work on washing salt. After undertaking the work on the laboratory scale, the work on bench scale using screw conveyor washery, disperse washing column and hydrocyclone has been carried out. Data on washing are published (Mohuiddin et al., 1966; Majeethia et al., 1984). The typical data are given in Table 4. In all the above work the objective was to obtain a Grade II salt for chemical industry as per I.S. 797-1982 (Table 5).

Due to advent of membrane technology for manufacture of caustic soda, the demand for high purity salt in India has now increased. Therefore, R&D work was undertaken to upgrade the freshly harvested salt to the quality as per B.I.S. Grade I salt for chemical industry. The present work provides the experimental results on washing freshly harvested and stored solar salt in a 30 kg/h bench-scale washery unit, established at the Institute.

EQUIPMENT USED AND PROCESS DETAILS

The washing unit consists of 2 screw conveyors, a gravity drainer and a vibrating drainer. The design parameters, viz. screw diameter, diameter to pitch ratio, revolutions of the screw and salt to wash liquor ratio and salt to fresh water ratio have been optimised.

After sieving, the graded raw salt is fed to the hopper of the first screw conveyor. Here it is washed with saturated brine having fixed magnesium ion concentration in a counter-current manner. The washed salt from the first screw conveyor is then fed to the second screw conveyor through a gravity drainer in which some wash-liquor is separated. In the second screw conveyor, the salt is further washed with plain water. The washed salt passes through the vibrating drainer where water is sprayed and then finally drained in the form of a heap. After 24–48 h draining, the final product is analysed. Part of the brine is rejected to maintain magnesium built up in the wash-liquor which is recycled. Figure 3 shows the schematic process diagram.

TABLE 4

Large-scale salt washing for ISI Grade II (Experimental salt farm)

Name of technique	Analysis of salt		% Removal of impurity
	Unwashed	Washed	
Washing in screw conveyor			
NaCl	98.00	99.17	–
Ca^{2+}	0.11	0.05	54
Mg^{2+}	0.46	0.14	69
SO_4^{2-}	0.82	0.32	61
Insolubles	0.12	0.05	58
Washing in disperse washing column			
NaCl	98.10	99.30	–
Ca^{2+}	0.08	0.04	50
Mg^{2+}	0.33	0.06	80
SO_4^{2-}	0.71	0.22	70
Insolubles	0.19	0.08	60
Washing in hydrocyclone washery (with centrifuge)			
NaCl	98.18	99.10	–
Ca^{2+}	0.31	0.20	36
Mg^{2+}	0.33	0.13	61
SO_4^{2-}	0.92	0.52	54
Insolubles	0.22	0.05	78

RESULTS

Experiments have been conducted to upgrade either freshly harvested salt from marine salt works or stored salt from marine and inland salt works. For each sample of salt three experiments were carried out and the average results of these three experiments are given in Table 5.

TABLE 5

Salt for chemical industries IS: 797-1982.

Sr. no.	Characteristic	Grade I	Grade II
1.	NaCl % by mass min.	99.5	98.5
2.	Matter insoluble in water % by mass, max.	0.05	0.20
3.	Calcium salts as Ca^{2+} % by mass, max.	0.03	0.20
4.	Magnesium salts as Mg^{2+} % by mass, max.	0.01	0.10
5.	Sulphates as SO_4^{2-} % by mass, max.	0.20	0.60
6.	Iron compound as Fe^{2+} ppm. max.	10.0	20.0

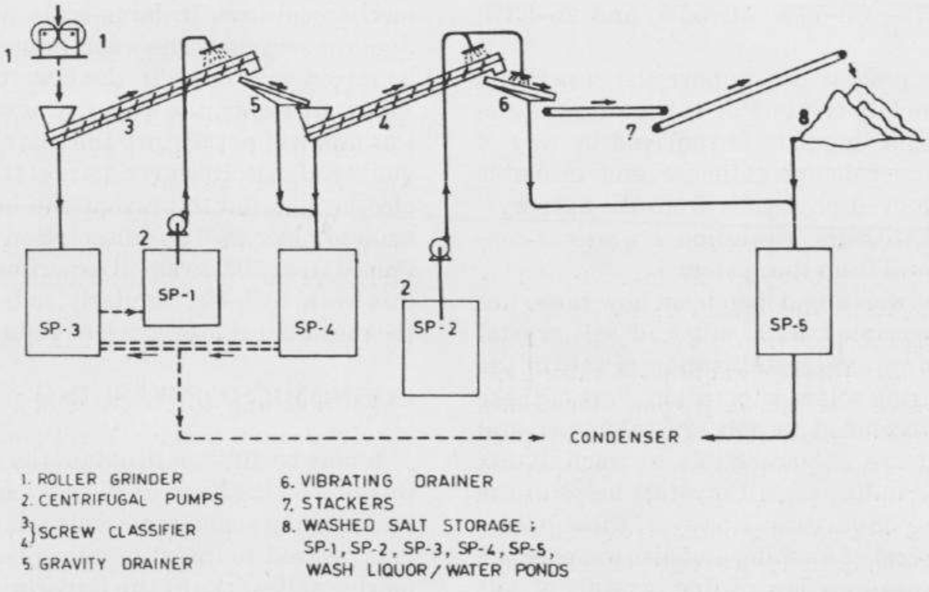

Fig. 3. Layout of the salt washing process.

TABLE 6

Results of bench-scale washing experiments

Sr. No.	Unwashed salt % composition					Washed salt on dry basis					Loss dissolution %	Loss mechanical %
	NaCl	Ca^{2+}	Mg^{2+}	SO_4^{2-}	Insolubles	NaCl	Ca^{2+}	Mg^{2+}	SO_4^{2-}	Insolubles		
1.	97.9	0.18	0.37	0.77	0.10	99.5	0.10	0.05	0.27	0.04	10	7.2
2.	97.6	0.14	0.46	0.80	0.12	99.4	0.06	0.07	0.25	0.05	10	5.0
3.	98.4	0.22	0.18	0.62	0.10	99.4	0.13	0.03	0.35	0.03	10	6.0
4.	96.5	0.32	0.57	0.92	0.16	98.3	0.23	0.18	0.60	0.08	10	6.0
5.	96.6	0.32	0.67	1.23	1.62	98.7	0.17	0.15	0.73	1.22	14	5.5
6.	95.7	0.21	0.55	1.16	1.35	98.0	0.11	0.21	0.51	0.63	15	6.0

Samples (1)–(3): Freshly harvested sea salt, Bhavnagar; (4): Freshly harvested well brine, Kharaghoda; (5)–(6): Stored well brine, Andhra Pradesh.
Impurity removal (%) — Samples (1)–(3): 44–57 Ca^{2+}, 84–86 Mg^{2+}, 43–81 SO_4^{2-}, 60–70 insolubles; (4): 28 Ca^{2+}, 68 Mg^{2+}, 34 SO_4^{2-}, 50 insolubles; (5)–(6): 46–47 Ca^{2+}, 61–77 Mg^{2+}, 40–56 SO_4^{2-}, 25–53 insolubles.

DISCUSSION

From the results given in Table 6, it can be seen that the upgrading of freshly harvested salt is better than that of stored salt. In the case of freshly harvested salt the purity of salt increases from 97.5–97.6% NaCl in raw salt to 99.4–99.5% NaCl in the final product. The calcium, magnesium, sulphate and insoluble matter are removed to the extent of 44–57%, 84–86%, 43–69%, and 60–70%, respectively. In the case of stored salt, upgrading by washing has been found to be less. The purity of raw salt from initial NaCl content of 95.7–96.6% has increased to 98.0–98.7% NaCl while calcium, magnesium, sulphate and insoluble matters have been reduced by 46–47%, 62–77%, 40–56%, and 25–53%, respectively.

The washing process can remove the superficial impurities from salt crystals or salt crystal aggregates. Magnesium impurity is removed by way of dissolution while calcium sulphate and insoluble matters are removed physically from the salt crystals and mixed with the wash-liquor which is continuously removed from the system.

The extent of washing depends on how these impurities are associated with salt and salt crystal aggregates during the crystallisation of salt in the crystallisers during solar evaporation. Part of these impurities are occluded in salt crystals or crystal aggregates and are not accessible to wash-liquor. Agitation and grinding of salt crystals helps in the removal of more impurities. However, these operations add to the cost of washing and also increase the loss of salt due to overflow of fine crystals of salt during washing. Therefore, in large-scale washing the solar salt is not ground but washed as such.

Occlusion of impurities during the manufacture of solar salt can be controlled by very strict density control of various ponds, maintaining proper depths in condensers and crystalliser and continuous flow of brine. Very large solar salt works in Australia and Mexico are able to obtain solar salt in which the occluded calcium and magnesium impurities are much less. Therefore, the removal of the impurities is improved by washing. Thus, the efficiency of the washing process can be increased by controlling the operational parameters during the salt manufacturing stage.

Loss of salt in the washing process is due to (i) dissolution of salt crystals in sea water/water and (ii) mechanical loss. In large-scale washing the wash-liquor is recycled. The wash-liquor after settling can be mixed with brine in the last stage condensers in solar salt works, the quantity of wash-liquor will be less and will not disturb the solar salt production or quality of salt. However, part of the wash-liquor can also be rejected to prevent the build up of magnesium impurities. Thus dissolution losses can be minimised from 10% in small experiments as reported in this work, to 3–4%. Similarly, in large-scale washing the mechanical losses can be reduced to 3–5%.

ECONOMICS OF WASHING

It may be difficult to obtain the exact economics of washing on the basis of 30 kg/h experiments without undertaking some large-scale trials on 1–2 t/h basis. It is desired to install a pilot plant washery at the marine salt works at the Experimental Salt Farm of the Institute to confirm the results of 30 kg/h wash-

TABLE 7

Estimated cost of mechanical washing with different capacity. Basis: (1) Washing plant is located in a salt works; cost of salt Rs. 100/T Rs. 1 lakh = Rs. 1,000,000; 20 Rs. = 100 Japanese Yen (approx.); (2) 200 working days at 20 h/day; (3) Direct labour Rs. 8/t; (4) Washed salt is stored for about 4–5 weeks before delivery; (5) Loss of salt (a) fines 6–7%, (b) dissolution 9–10%, (c) Handling 4–5%, total estimated 22% (considering 70% recovery from fines and dissolution nett loss will be 10%)

	Capital investment	Capacity (t/y)		
		5 t/h (20,000)*	10 t/h (40,000)*	15 t/h (60,000)*
1.	Equipment cost	7.00	10.75	15.25
2.	Add 2% installation charges	1.40	2.15	3.15
3.	Civil works	0.88	1.66	2.44
	Total	9.28	14.56	20.84
	Say	9.30	14.60	20.90
	Process cost:			
1.	Raw materials			
	22% (a) Salt loss tonnes	4.4 (4,400)	8.8 (8,800)	13.2 (12,300)
2.	Utilities			
	(a) Electricity, kWh	1.55 (124,000)	2.8 (224,000)	4.0 (320,000)
	(b) Water, kl.	0.10 (5,000)	0.20 (10,000)	0.30 (15,000)
3.	Indirect labour	0.25	0.50	0.50
4.	Direct labour	1.60	3.20	4.80
5.	Depreciation			
	(a) 20% on P&E	1.68	2.58	3.68
	(b) 10% on civil works	0.09	0.17	0.24
6.	Maintenance			
	(a) 10% on P&E			
	(b) 5% on civil works	0.88	1.38	1.97
7.	Rent, taxes 2% on total capital	0.19	0.29	0.41
8.	Interest 18% on total capital	1.67	2.63	3.76
	Total cost of washing	12.41	22.55	32.86
	Cost of washing (Rs./t of salt)	62.05	56.40	54.80

*(Rs. in Lakhs).

ing unit. This depends upon the availability of funds. However, the pre-design cost estimates for 5, 10 and 15 t/h washery units are estimated. The cost of washing for these capacities under the present price is calculated to be Rs. 62, Rs. 56.4 and Rs. 54.8 per tonne of salt (Table 7).

SAVINGS IN CHEMICAL TREATMENT COST ON WASHING

When the raw solar salt is subjected to washing, calcium, magnesium and sulphate impurities are reduced. Because of this there will be a difference in the cost of chemicals used for treating the brine in both soda-ash and caustic soda industries. Taking into consideration the maximum impurities levels found in salt, nomograms are prepared for arriving at the quantities of chemicals required for brine purification. (Figs. 4 and 5).

The experimental results indicate that, on average, 45% of calcium, 80% of magnesium and 60% of sulphate impurities can be removed by subjecting freshly harvested salt to the simple washing technique mentioned here. Taking a typical example of raw salt and washed salt, the chemical treatment cost has been calculated and is given in Table 8. It will be seen that the saving in the cost of chemicals, which also considers the cost of washing, is about 40%. Thus washing of salt not only saves the cost of chemicals utilised for chemical treatment for preparing process brine and soda ash and caustic soda industry but also helps indirectly by reducing the bulk of sludge [$Mg(OH)_2$, $CaCO_3$ and $BaSO_4$] formed

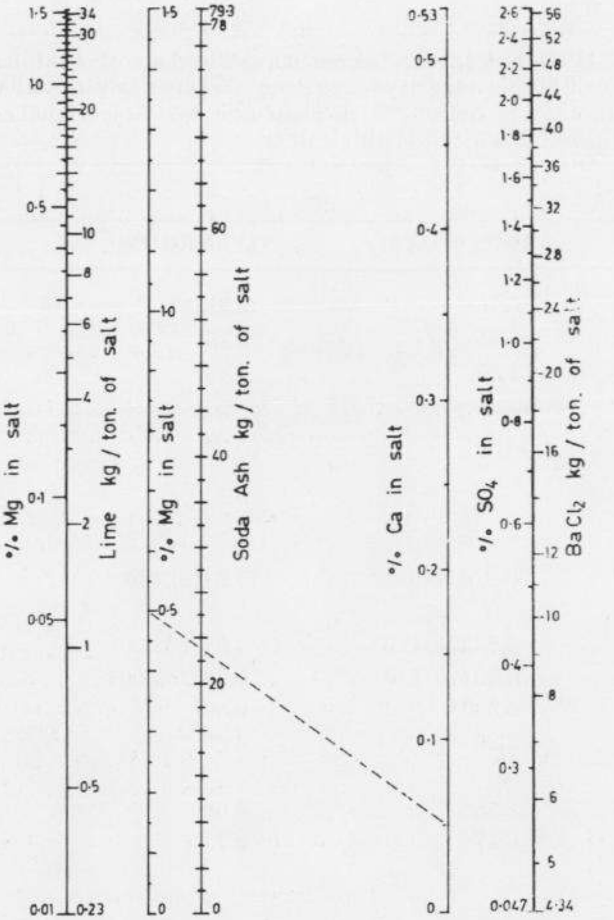

Fig. 4. Nomogram: chemicals required for removal of impurities from salt.

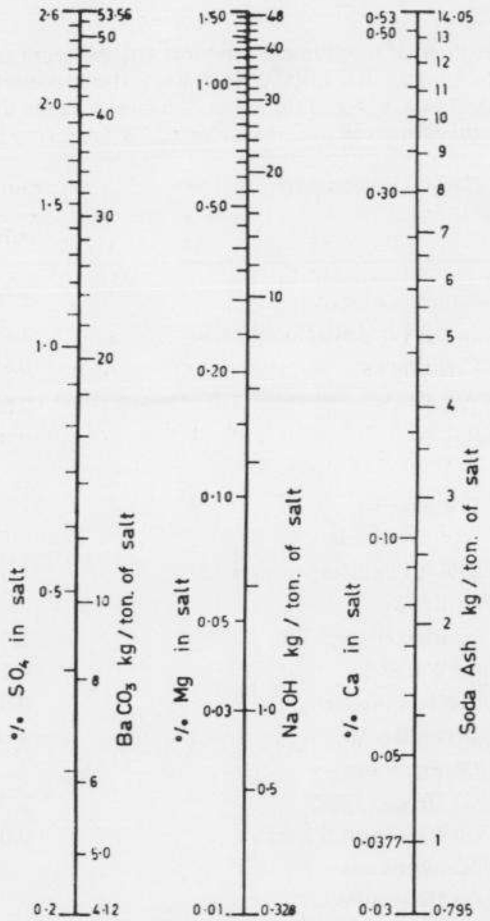

Fig. 5. Nomogram: chemicals required for removal of impurities from salt.

TABLE 8

Cost of chemicals for unwashed and washed salt. Basis: (1) Impurities % — initial: 0.15 Ca^{2+}, 0.20 Mg^{2+}, 0.80 SO_4^{2-}; final: 0.083 Ca^{2+}, 0.04 Mg^{2+}, 0.36 SO_4^{2-}. (2) Cost of chemicals Rs./t. 70% lime 600, barium chloride 16,000, soda ash 5,000, barium carbonate 14,000, caustic 11,000

	Cost of chemicals Rs. /T of Salt			Washing cost (Rs./t)	Total cost (Rs./t)
	Soda ash	Lime	Barium chloride		
(A)					
Unwashed salt	63.48	3.94	277.76	–	345.18
Washed salt	19.69	0.78	125.00	62.05	207.52
				% Saving	40.00
	Soda ash	Caustic	Barium carbonate		
(B)					
Unwashed salt	19.88	72.16	230.72	–	322.76
Washed salt	11.00	14.43	103.82	62.05	191.13
				% Saving	41.00

during the treatment. It can also help in reducing the size of equipment used for chemical treatment.

CONCLUSIONS

Washing of salt is a simple and low cost process to upgrade solar salt. Washing of freshly harvested salt yields high purity salt as required by the caustic soda industry utilizing mercury and membrane cell technology. The amount of sludge produced during the chemical treatment of brine is reduced by about 30–50% which reduces the problem of its disposal. Due to the reduction in the bulk of the sludge, the mercury entrapment is reduced from which mercury can be recovered and may reduce the mercury pollution.

Large-scale salt manufacturers can afford to use mechanical washing for improving the quality of solar salt in India.

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