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EFFECTS OF AGITATION AND BITTERN QUALITY ON RECOVERY OF MAGNESIUM SULPHATE

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Experiments have been conducted in an externally cooled batch chiller (crystallizer) to study the effects of (i) agitation level, (ii) stirrer design and (iii) quality of sea bittern, on the recovery of magnesium sulphate and other variables like heat flow rate, heat of crystallization and heat transfer coefficient. The studies indicate that the linearity of temp-time plot and its slope are neither affected by the agitation level nor by quality of sea bittern (excepting spent sea bittern). Recovery/crystallization patterns (or conc-temp relationships) have been found critically dependent on agitation level, degree of turbulence, stirrer design and bittern quality. Three straight blades stirrer is found to be more suitable whereas, 6 curved blades stirrer causes greater turbulence thereby resulting into reduced recovery. The optimum level of agitation is found to be around 165 r p m (tip velocity 62.5 m min^{-1} and Reynolds number 13000), around which incidently, both, (a) percent recovery against temperature and (b) energy consumed in stirring, Kj min^{-1} Vs Reynolds number behave linearly, the recovery is at its optimal, and the energy consumption is minimum. Beyond the optimal limit of (165 r m p) 62.54 m min^{-1} tip velocity, the performance parameters become independent of agitation rate, however, under such situation they are influenced by the quality of sea bittern and hence found to change unpredictably. Normal sea bittern is found to be a suitable raw material, whereas any pretreatment of normal bittern renders it unsuitable and at times an uneconomical raw material.

Key words: Sea bittern, Magnesium sulphate, Heat of crystallization, Heat transfer coefficient.

Introduction

The original studies concerning the recovery of magnesium sulphate during chilling of two types of sea bittern (normal and heated) mainly comprised the investigations of concentration drop and percent recovery as function of temperature and the statistical analysis of the data (Beg *et al* 1989; Nayeemuddin and Mahmood 1991). Subsequently, based on (a) linearity of (i) temperature-concentration, (ii) temperature-time plots and (b) the modified energy balance equation, relationships were derived for calculating the values of the thermal/thermodynamic parameters like heat of crystallization, rate of heat flow (Removal) from the system and heat transfer/utilization coefficients (Nayeemuddin and Mahmood 2000). In the original work (Beg *et al* 1989), the data lacked the quantitative effects of rate of agitation on the recovery of magnesium sulphate and other parameters, as, therein agitation level remained constant. Therefore, in order to fill in the gap, experiments have been arranged in a simple jacketted chiller in which level of agitation/degree of turbulence could be varied either by changing the agitation rate or stirrer design, (shape/number of blades). Availing the opportunity, the influence of bittern quality has also been investigated comprehensively. The effects of agitation level, stirrer design and bittern quality on different parameters have been explained. Further, useful data in respect of average crystallization temperature of magnesium sulphate against its average initial percentage in original sea bittern samples, has also been studied.

Materials and Methods

The raw materials comprised three types of samples namely (i) normal sea bittern, density ranging from 1.29 to 1.32 gml^{-1}, from which sodium chloride of even commercial purity could not be recovered anymore, (ii) spent sea bittern, from which magnesium sulphate had already been recovered by chilling and the residual liquor was subsequently solar evaporated to the desired density to enhance the concentration level of magnesium sulphate and (iii) lean bittern sample, reconstituted with magnesium sulphate to increase its concentration level almost equal to the normal bittern. Two stirrers of standard design (McCabe and Smith 1985) viz 6 curved and 3 straight blades were used at three r p m levels 370, 218 and 165.

The method consisted of charging each time 36.2 kg of sample to the chiller, a suitably insulated vessel complete with appropriate cooling and stirring systems having independent ar-

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rangement for taking out samples through a syphon and measuring the temperature accurately (± 0.5°C) using a mercury thermometer. The vessel was provided with conical bottom to facilitate the removel of magnesium sulphate on completion of each experiment. During chilling, temperature was noted and samples were syphoned off after definite time intervals. Collected samples were filtered immediately under vacuum; definite amount of filtrate was pipetted out, weighed and analysed chemically.

In the absence of relavent data concerning the conversion of mechanical work into heat in the specific case of stirring of sea bittern, a pragmatic approach was adopted for measuring the actual power consumed (at different agitation levels) by the electrical motor in stirring sea bittern, density 1.24 gml^{-1} to 1.32 gml^{-1} and viscosity 3.8 to 4.2 c p around 30°C. An electronic invertor was used to set the agitation at any desired level which also indicated voltage, amperage and wattage of stirrer at any moment of operation. The data so collected explained appropriately the conversion of mechanical work into heat.

Data representation. (i) Design/performance data. Essential data in respect of chiller performance, raw material, agitation level and Reynolds number is shown in Table 1.

(ii) Time-temperature-concentration data: Pertinent data collected in respect of different sets of experiments is shown by temp-time graph (Fig 1) and the concentration (magnesium sulphate)-temperature plots Figs (2, 2a).

(iii) Thermal and related data: Basic data like cooling range, average temperature of crystallizing slurry, the overall average temperature drop between crystallizing slurry and evaporating refrigerant, along with the average values of the slopes of the plots of temp-time and conc-temp calculated both from the graph and by regression; is represented in Table 2.

(iv) Percent recovery-temperature data: Percent recovery of magnesium sulphate estimated as function of temperature for different types of samples of sea bittern/agitation level indicated in Fig 3.

(v) Thermal/thermodynamic data: Pertinent data namely average specific heat, heat of crystallization, rate of heat flow, heat transfer/utilization coefficient estimated by various relationships, derived previously (Nayeemuddin and Mahmood 2000) along with intial percentage of magnesium sulphate/chloride, is appended in Table 3.

(vi) Average crystallization temperature of magnesium sulphate and its corresponding initial concentration: Concentration-temperature curve does not give fair idea concerning initial crystallization temperature of magnesium sulphate because of initial non-equilibrium conditions. However, as pointed out earlier (Beg *et al* 1989; Nayeemuddin and Mahmood 1991; Nayeemuddin and Mahmood 2000), the intercept of the temperature-time and percent recovery-temperature plots give fairly accurate idea of crystallization temperature of magnesium sulphate for its given initial concentration. Accordingly, average values of the crystallization temperature in respect of different samples of sea bittern have been determined (both by regression and graph) and plotted against corresponding initial average concentration of magnesium sulphate in Fig 4.

(vii) Conversion of mechanical work into heat: In order to assess the mode of change of the mechanical work into heat, the data collected in respect of electrical energy consumed due to stirring calculated in 5 Kj min^{-1} using appropriate formulae is plotted against the Reynolds number (Fig 5). The curve so obtained has been appropriately explained.

Results and Discussion

As for the temp-time plots (Fig 1), separate lines in each experiment are indicative of linear relationship with high order of correlation coefficient (above 0.99 in all cases). The intercept (on y-axis) represents average value of crystallization temperature corresponding to the initial concentration of magnesium sulphate of given sample, whereas its slope represents the rate of temperature drop and is instrumental in

Table 1
Essential design features and performance data of the chiller

Design features	Performance
Net capacity	28 lit
Diameter of chiller	30 cm
Cooling area	0.383 m^2
Compressor rating	0.5 KW ~ 0.75 hp
Refrigerant	Feron - 12
Evaporation temp of the refrigerant(t_s)	-15°C
Suction pressure	12 psi
Maximum installed cooling capacity	9045 Kj h^{-1}
at operating conditions (H)	=150.75 Kj min^{-1}
Other data:	
In respect of raw material (Sea bittern)	
Mass	36.2 kg batch^{-1}
Density	1.29 to 1.32 gm l^{-1} at 30°C
Viscosity	3.8 to 4.2 CP (30°C)
Data in respect of agitation:	
Average diameter of agitator	12.06 cm

Table 1a

Linear velocity of the tip of stirrer and Reynolds number as function of agitation level.

R.P.M.	Tip velocity* of stirrer m min^{-1}	Reynolds* number
370	140.24	1,03,000
218	82.62	55,000
165	62.54	43,000

*In the broader context of scale-up design [1], merely agitation level does not carry much sense, rather velocity of tip and Reynolds number are useful parameters.

calculating the operating parameters (Nayeemuddin and Mahmood 2000) (viz rate of heat flow, heat transfer/utilization coefficient etc). The slopes of different sets of experiments show abrupt change with respect to agitation rate/ Reynolds number, being lowest at 218 rpm (spent sea bittern) and more close but higher values at 370 rpm (normal bittern) and 165 rpm (reconstituted sea bittern). This clearly suggests that at least beyond 165 rpm or, tip velocity 62.5 $mmin^{-1}$ and Reynold number 43000, rate of agitation does not influence the rate of temp drop/rate of cooling and perhaps it is the quality of sea bittern which becomes a dominating factor.

The examination of conc-temp plots (Fig 2 and 2a) in the light of the quality of sea bittern and the rate of agitation, brings the following to the surface:

The nonlinear portion(s) at the inception of experiment in different situations are probably because of the initial non-equilibrium conditions. Unprecedented case of two nonlinear portions, one immediately followed by the other but in opposite directions in the instance of reconstituted sea bittern, depicts the initial high rate of crystallization immediately followed by the lower one. Two separate curves (Fig 2) with slight difference in slopes for the same raw material and agitation level (370 rpm), but for two different stirrer designs (3 and 6 blades) suggest that both high agitation level and degree of turbulence affect the crystallization patterns and cause the curves to become asymtotic to the temperature axis during last stages in low temperature-concentration regions. This also indicates that irrespective of stirrer design, the agitation level of 370 rpm (tip velocity 140.25 $mmin^{-1}$) is sufficient to bring about a sort of pseudo equilibrium by suppressing further crystallization in low temperature-concentration regions.

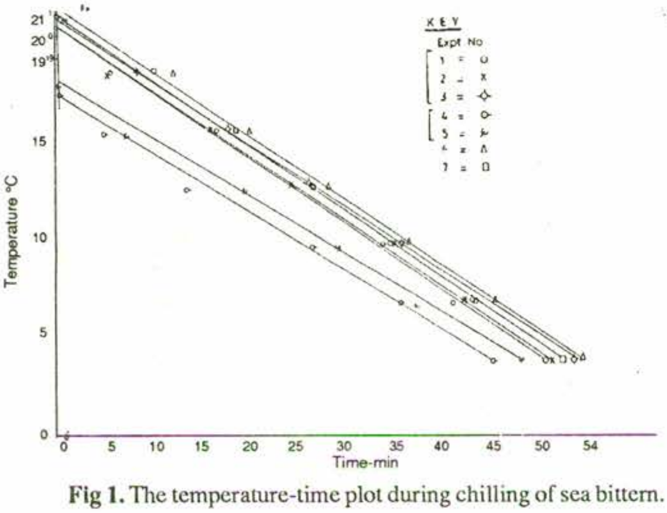

Fig 1. The temperature-time plot during chilling of sea bittern.

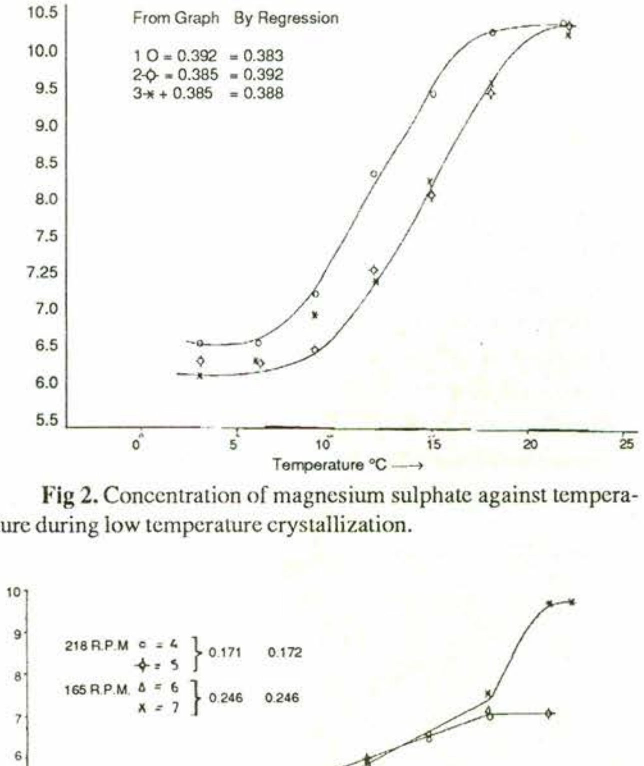

Fig 2. Concentration of magnesium sulphate against temperature during low temperature crystallization.

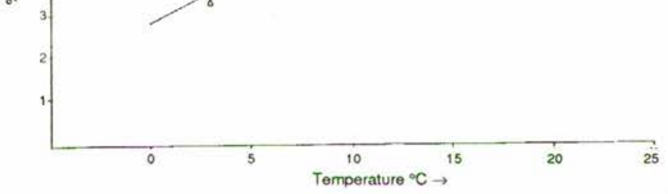

Fig 2a. Concentration of $MgSO_4$ against temperature during low temperature crystallization.

The slope of the linear portion (Table 2, Fig 2 & 2a) which represents the quantity of magnesium sulphate crystallized per degree fall of temp per unit mass of bittern is characteristic of the raw material and suggests that pretreatment of normal sea bittern has direct bearing on the slope, which in turn accordingly affects the heat of crystallization. Projection of the linear portions of conc-temp plots (Fig 2 & 2a) on x-axis represents the temperature range within which the previously derived relationships (Nayeemuddin and Mahmood 2000) are applicable.

Examination of the plots (Fig 2 & 2a) indicates that the temp range (Table 2) is contained by higher agitation level particularly 370 and above; this in turn affects to some extent average and overall average temperature drop.

Examination of percent recovery temperature curves (Fig 3) in context with the influence of rate of agitation, reveals that: (a) The tip velocity of 140.24 m min^{-1} (370 rpm), irrespective of stirrer design, causes curves to become asymtotic to temperature axis, thereby restricting recovery to 38-40% in low temperature concentration regions. Further, two separate curves for different stirrer designs but with the same agitation level (370 rpm) suggest that degree of turbulence also governs recovery crystallization pattern. (b) In case of tip velocity 82.62 mmin^{-1} (218 rpm), the curves appear tending to be linear and (c) at tip velocity 62.54 mmin^{-1} (165 rpm), the relationship between variables is linear. As for the influence of the quality of sea bittern on recovery, it is lowest (35.6 to 38.59%) in case of spent sea bittern suggesting that the use of this raw material would be uneconomical. The recovery is highest in the case of reconstituted sea bittern which may partly be attributed to the characteristic of raw material and partly to low agitation rate (165 rpm). Low recovery in case of normal bittern, as already explained, is only because of high degree of turbulence. Two separate curves for each of the two instances of pretreated samples of spent and reconstituted sea bittern substantiate that percent recovery is also very sensitive to composition/quality of sea bittern.

Values of specific heat (Table 3) of various samples determined from the percentage composition of the components (Perry 1950) and their respective specific heat data are found to be very close. It is likely that the composition of the components in various samples vary in such a fashion that the numerical values of the parameter in different situations do not show much variations. In the case of heat of crystallization for normal sea bittern, values are comparable to the previous one (Nayeemuddin and Mahmood 2000). Pretreated samples show the enhanced values of the heat of crystallization, 40% to 50% increase in case of reconstituted sea bittern and 100% for spent sea bittern, thereby increasing the cooling cost.

As for the operating parameters, review (Table 3) shows that their values agree well within the same type of bittern samples, whereas the data pertaining to different quality bitterns do not agree. Analysis of numerical values of the parameters (Table 3) shows that rate of cooling and heat utilization coefficient, estimated from slope of temp-time plot, reflect the same irregularity with reference to agitation level as in the case of slope itself viz lowest values at 82.62 mmin^{-1} (spent sea bittern) and higher and close values for normal bittern (140.24 mmin^{-1}) and reconstituted sea bittern (62.5 mmin^{-1}). Further considerations reveal that in case of spent sea bittern, firstly

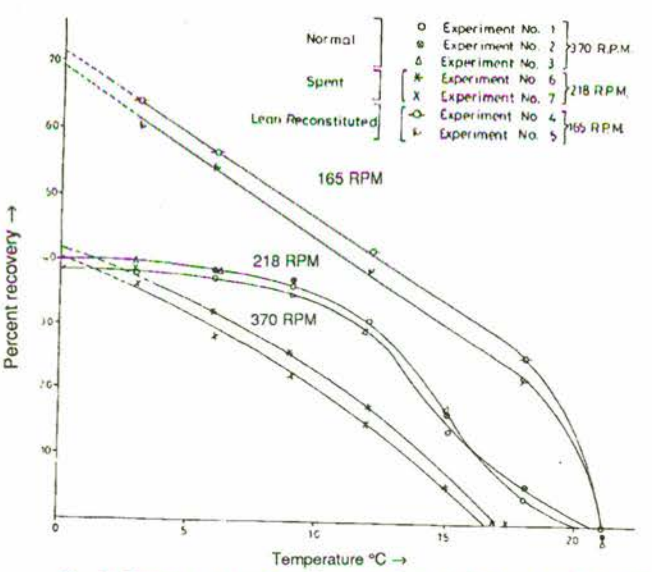

Fig 3. Percent recovery of magnesium sulphate against temperature.

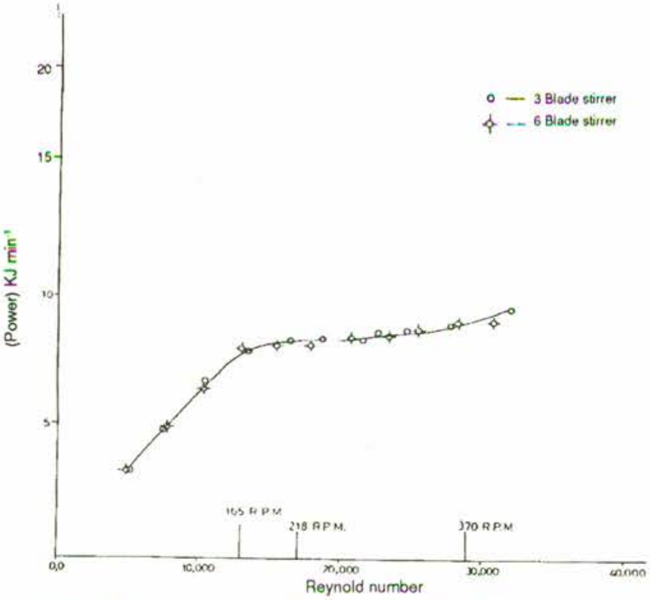

Fig 4. Energy consumed in stirring against N_{Re}.

Table 2

Operating/thermal data during chilling of sea bittern for different sets of experiments

Exp No	No of blades	RPM	*Temp range of cooling (°C)	t_{ave} (°C)	Δt_{ave} (°C)	Slope (m_{c-t}) conc-temp plot			Slope (m_{t-t}) temp-time plot		
						From graph	By regression x 10^{-2}	Average	From graph	By regression	Average
1	3	370	6.5	12.25	27.25	0.392	0.383	0.387	0.327	0.326	0.326
2	6	370	7.0	14.5	29.5	0.385	0.392	0.388	0.325	0.324	0.320
3	6	370	7.0	14.5	29.5	0.385	0.383	0.386	0.325	0.318	0.321
4	3	218	15	10.5	25.5	0.171	0.172	0.171	0.295	0.282	0.288
5	3	218	15	10.5	25.5	0.171	0.172	0.171	0.289	0.290	0.289
6	3	165	15	10.5	25.5	0.246	0.246	0.246	0.321	0.318	0.319
7	3	165	15	10.5	25.5	0.246	0.246	0.246	0.326	0.319	0.322

*The temp range within which con-temp plot maintains linearity.

Table 3

Performance parameters

Expt No	Type of sea bittern	Specific Heat KJ Kg^{-1} °C (Cp)	Heat of crystallization KJ Kg^{-1} of magnesium crystallized sulphate $\lvert -H_c \rvert^x$	Heat transfer coefficient KJ min^{-1} m^2 °C U;	Rate of heat removal/cooling KJ min^{-1} (Qc)	Heat utilization coefficient (C_o)	Composition of sea bittern	
							% $MgCl_2$	%$MgSO_4$
1	Normal	3.06	789.68	6.92	72.22	0.479	13.87	10.2
2	Normal	3.06	787.64	6.28	71.00	0.471	13.90	10.2
3	Normal	3.07	794.31	6.32	71.45	0.474	14.13	10.18
4	Spent$^+$	3.00	1749.85	6.42	62.68	0.416	19.68	6.97
5	Spent$^+$	3.04	1769.68	6.51	63.61	0.422	19.64	6.96
6	Reconstituted^o	3.07	1247.96	7.34	71.68	0.475	16.11	9.65

$\lvert -H_c \rvert^x$ Indicates the numerical value of heat of crystallization; + The samples from which magnesium sulphate already recovered by chilling and resulting liquid was subsequently solar evaporated to the desired density to enhance percentage of magnesium sulphate; ^{o} By adding extra magnesium sulphate to enhance its percentage to the desired level.

the increased values of heat of crystallization lower down the rate of temperature drop/cooling rate. Secondly, perhaps the increased concentration of magnesium chloride (19.5%) and reduced percentage of magnesium sulphate (7.2%), enhance the viscosity of the slurry thereby increasing the thermal resistance and decreasing the rate of cooling and heat utilization coefficient, which suggests that the quality of sea bittern is a governing factor. Heat transfer coefficient shows just inverse values with respect to rate of agitation viz lower values (6.28 to 6.92 kjmin^{-1}) for higher agitation rate (370 and 218 rpm) and higher values (7.34 kjmin^{-1}) for lower agitation rate (165 rpm). Probably the cause of these rotated values may be that the parameter in addition to the rate of heat flow also depends on overall average temperature drop (Table 2), which in turn, as pointed earlier, to some extent is affected by agitation level; nevertheless, quality of sea bittern remains a deciding factor. Generally the operating parameters are not exclusively governed by the characteristics of raw material and rather agitation level/Reynolds number, remains a dominating factor. However, as pointed out previously, perhaps beyond certain Reynolds number (agitation rate) these parameters become independent of agitation level. It therefore transpires that 165 rpm (tip velocity of 62.54 mmin^{-1}) in the present case is greater than such a limit and consequently characteristic of raw material/quality of bittern becomes a dominating factor.

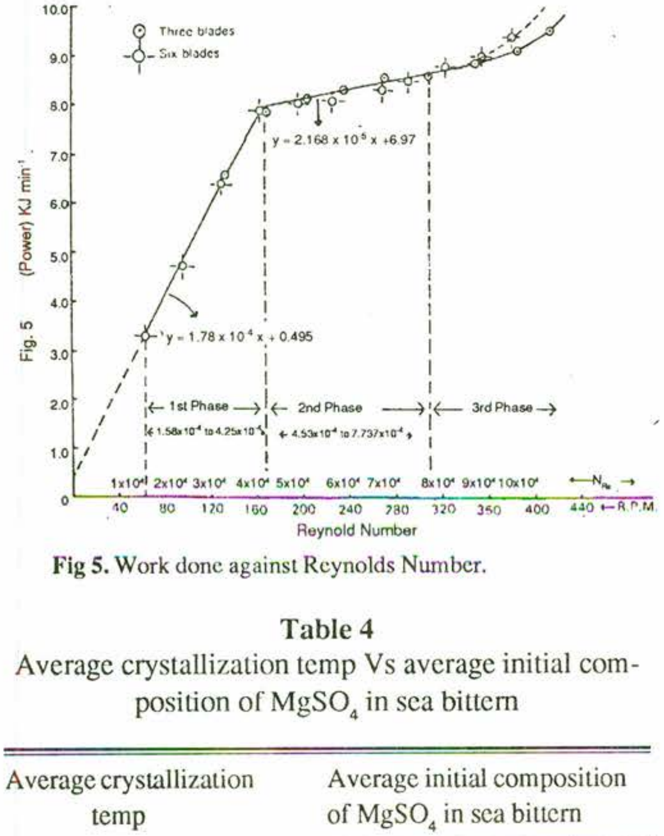

Fig 5. Work done against Reynolds Number.

Table 4

Average crystallization temp Vs average initial composition of $MgSO_4$ in sea bittern

Average crystallization temp	Average initial composition of $MgSO_4$ in sea bittern
1. 20.85°C	9.96
2. 19.89°C	7.91
3. 17.28°C	6.975
4. 16.03°C	6.157
5. 13.9°C	5.48

As in evident from Fig 4, relationship between variables viz average, crystallization temp of magnesium sulphate in sea bittern and the average initial concentration of magnesium sulphate, is linear upto 20°C and nonlinear between 20°C and 21°C, beyond which the curve becomes asymtotic to temperature. The average concentrations of magnesium sulphate corresponding to 20°C and 21°C are around 8.4% and 10% respectively; the latter one being the limiting or saturated concentration of magnesium sulphate. Although Fig 4 is simple one, but it is more useful as it explains the crystallization pattern of magnesium sulphate from bittern solution.

Analysis of conversion of mechanical work into heat. The plot (Fig 5) showing the work done in stirring (Kjmin^{-1}) against Reynolds number comprises three portions; the first (r=0.998) and second one (r=0.972) being linear and third one non-linear. The algebric equations for the first two linear phases, and the range of N_{Re} within which the said relationships are applicable have been indicated in Fig 5.

The intercept on y-axis of the first phase would be the amount of energy (0.695 Kjmin^{-1}) to overcome the inertia of the system; viz if the energy supplied is less than the said value, the stirrer shows no movement at all. The start of the second phase and the termination of the first one is indicated by sudden reduction in slope, resulting into fall in energy with N_{Re}. This occurs at about 1658 pm around^{-1} N_{Re} 43000 which incidently is the lowest chosen agitation level whence perhaps the rate of energy spent in stirring overcomes the inertial forces. Almost at initiation of the third phase the curves for 'three and six blades' which were almost the same upto N_{Re} 8.00 x 10^4, take separate paths suggesting that beyond the said N_{Re} extra turbulence for the same agitation level/N_{Re} needs more energy.

As for the conversion of mechanical work into heat, it transpiers from the above considerations and the pattern of the curve that during first phase there appears a little probablity of changing the mechanical work of stirring into heat. Nevertheless, during second and third phases, a portion of differential energy (1.5 Kjmin^{-1}) required for increasing N_{Re} from 4.3 x 10^4 to 10.3 x 10^4 may change into heat energy. Even if it is assumed that the whole of the differential energy of 1.5 Kjmin^{-1} changes into heat, it may for all practical purposes be ignored in comparison with high order of rate of heat removal (62 to 72 Kjmin^{-1}) form the system, without causing any significant error in the estimated data concerning thermal/thermodynamic parameters.

Conclusion

The studies show that neither quality of sea bittern nor agitation level and degree of turbulence ($N_{Re} \sim 103500$) affect the linearity of temp-time plots which otherwise show high order of correlation coefficient (above 0.99). In case of conc-temp plots it is observed that rate of agitation/degree of turbulence, stirrer design and quality of sea bittern affect crystallization/recovery patterns of magnesium sulphate during its low temperature crystallization from sea bittern. High agitation levels, (N_{Re} around 1, 03,000), irrespective of stirrer design suppress further crystallization or bring about a sort of pseudo equilibrium in low temperature concentration regions thereby resulting into reduced recovery. The degree of turbulence caused by stirrer design (six curved blades, 370 rpm) also affects slightly the slope of the conc-temp plot.

Further, the studies suggest that optimum tip velocity is 62.54 mmin^{-1} (N_{Re} 43000 165 rpm) around which the inertial forces are almost overcome, the relationships between (I) energy consumed by the stirrer (Kjmin^{-1}) against Reynolds number and (II) the percent-recovery-temperature, remain linear, recovery is at its optimal, the energy consumption is low and the chances of suppression of crystallization are marginalized. The agitation rate higher than 62.54 mmin^{-1} tip velocity (165 rpm) causes the parameters, like the slope of temperature-time plots/the rate of temperature drop, rate of heat removal from the system (rate of cooling) and heat transfer/utilization coefficient, to become independent of agitation rate. Consequently bittern quality becomes a dominating factor which in turn affects the above variables abruptly.

It is observed that normal sea bittern is the most appropriate raw material for the recovery of magnesium sulphate from sea bittern. The pretreatment of sea bittern decreases the value of slope of conc-temp plot, thereby increasing the numerical value of the heat of crystallization which results into increased processing cost. Spent sea bittern is uneconomical because of its low recovery and higher operating (cooling) cost. The recovery in case of reconstituted bittern is the highest but cooling cost would be higher than normal bittern and moreover it shall add one more step to production schedule without much benefit.

The relationship between the average crystallization temperature and initial concentration of magnesium sulphate in sea bittern is found to be linear upto 20°C, nonlinear between 20°C and 21°C and thereafter becomes asymtotic to temperature axis. The saturated concentration of magnesium sulphate in sea bittern around 21°C is about 10%. This suggests that during the recovery of sodium chloride, crystallization of magnesium sulphate particularly in last stages, could be suppressed by maintaining the temperature of bittern around 20°C to 21°C.

Notations.

C = Any concentration attained corresponding to temperature
C_f = Final concentration or weight fraction of $MgSO_4$ in solution Kg of $MgSO_4$ 100 Kg^{-1}sol.
C_i = Initial concentration or weight fraction of $MgSO_4$ in solution Kg of $MgSO_4$ 100 Kg^{-1}sol.
ΔC = Drop in concentration during chilling ($C_i - C_f$)
C_p = Ave. specific heat of the liquid bulk KJKg^{-1} °C
D_a = Diameter of agitator, m
C_o = Heat utilization coefficient
H_c = Heat of crystallization KJ Kg^{-1}$MgSO_4$ crys.
H = Rated cooling capacity of chilling unit in KJh^{-1} or KJmin^{-1}
n = Number of revolution min^{-1} (rpm)
t_i = Initial temp °C
t_f = Final temp °C
Δt = (t_i - t_f) temp drop during chilling °C
t = Any temperature during chilling °C
C = Correlation coefficient
t_s = Condensing temp of refrigerant °C
t_{ave} = Average temp of crystallizing slurry °C $(t_i + t_f)2^{-1}$
Δt_{ave} = Overall average temp drop between crystallizing slurry and evaporating refrigerent ($t_{ave} - t_s$)
U_i = Overall average heat transfer co-efficient based on inside area of chiller
θ_t = KJ h^{-1} m^{-2} °C = KJ min^{-1} m^{-2}
θ_f = Final time of chilling in min
$\Delta\theta$ = Difference in initial and final time
ρ = Density of liquid gml^{-1}
μ = Viscosity of liquid poise (gem cm^{-1} sec^{-1})
$m_{c\text{-}t}$ = Slope of conc-temp plot
$m_{t\text{-}t}$ = Slope of temp-time plot
N_{Re} = $\dfrac{\pi D_a^2 n\rho}{\mu}$ (Reynolds number)

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