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PREPARATION OF SODIUM SULPHATE FROM ROCK SALT AND AMMONIUM SULPHATE

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Investigations were carried out for the preparation of anhydrous sodium sulphate (salt cake) from rock salt (NaCl) and ammonium sulphate (commercial grade). Effect of temperature, time of reaction and varying proportion of the reactants were studied and parameters were established for the maximum recovery of sodium sulphate. The purity of the product (Na_2SO_4) has been found to be 98% with a recovery of 95%.

Key words: Preparation, Rock salt, Sodium sulphate.

Introduction

The preparation of anhydrous sodium sulphate from sodium chloride and sodium bisulphate has long been known [1]. The sodium bisulphate for the purpose is either being derived from the manufacture of nitric acid by the action of sulphuric acid on chile salt petre (sodium nitrate), or is obtained by the action of sulphuric acid on common salt.

Sodium sulphate could also be produced as a by-product during the production of hydrochloric acid from sodium chloride and sulphuric acid [2]. This process is however, of insignificance due to high cost of production. A process of some importance for the preparation of sodium sulphate is the Hargreave-Robinson Process [3] whereby sulphur dioxide, air and steam are passed over salt granules.

Volfkovich *et al.* [4] studied the use of roasted pyrite as a catalyst for the preparation of sodium sulphate from sodium chloride and sulphur dioxide and found that at the optimum temperature of 500-5°, the use of 1% of pyrite decreases the time required for 95-98% conversion of sodium chloride to sodium sulphate from 16-20 days to a matter of hr. H. Baur [5] melted an intimate mixture of cryolite and gypsum and extracted sodium sulphate by leaching the mass with water.

The use of mixture of sodium chloride with copper sulphate [6], Zinc sulphate [7], manganese sulphate [8] and lead sulphate [9] have also been patented as processes for converting sodium chloride into sulphate. None of these methods are economical and viable due to high cost of the reactants and necessitates complicated unit operations in producing sodium sulphate.

Sodium sulphate obtained by the method undertaken would be economical as the reactants are readily available and inexpensive. The process involved is also simple and do not require any complicated operational units. The raw materials i.e. sodium chloride and ammonium sulphates are abundantly available within the country.

Materials and Method

Chemical composition of raw materials. The chemical analyses of the reactants i.e. sodium chloride and ammonium sulphate has been undertaken [10] and their percent composition is shown in Tables 1,2.

PREPARATION OF SODIUM SULFATE

Procedure. Rock salt of 30 mesh size and ammonium sulphate are mixed thoroughly in a mortor and placed in a porcelain boat. The boat is then heated electrically in a tube furnace. The ammonium chloride formed as a result of the interaction of the two reactants sublimes whereas sodium sulphate is obtained as indicated by the following reaction

$$(NH_4)_2SO_4 + 2NaCl \longrightarrow Na_2SO_4 + 2NH_4Cl.$$

The sodium sulphate so obtained is then dissolved in water and its purity is determined. Effect of different parame-

TABLE 1. COMPOSITION OF ROCK SALT (NaCl).

Water insoluble matter	=	9.80%
Sodium as Na^+	=	33.40%
Chlorides as Cl	=	51.50%
Calcium as CaO	=	1.40%
Sulphates as SO_3	=	1.30%
Magnesium as MgO	=	0.50%
Potassium as K_2O	=	1.40%
Carbonate and bicarbonate	=	0.30%

TABLE 2. COMPOSITION OF AMMONIUM SULPHATE $(NH_4)_2SO_4$.

Water insoluble matter	=	1.20%
Nitrogen as NH_3	=	25.30%
Sulphates as SO_4	=	72.40%
Calcium as CaO	=	0.30%
Magnesium as MgO	=	0.10%
Sodium as Na_2O	=	0.30%
Potassium as K_2O	=	0.20%

ters like temperature, time of reaction and stoichiometric ratios were also studied.

Effect of temperature. The reactants were heated at different temperatures and the results are shown in Table 3. It has been found that maximum conversion of sodium chloride and ammonium sulphate occurs at a temperature of 350° to produce sodium sulphate with a recovery of 95%.

Effect of time. The reactants were heated in the tube furnace for different time interval and the findings are shown in Table 4. It was found that better results are obtained when the reaction is undertaken for a period of 1 hr. at a temperature of 350°.

Stoichiometric ratio (w/w). Different ratios of the reactants were studied under the optimum conditions of temperature and time as mentioned above. The results are shown in Table 5. It was found that a ratio of 2:1 NaCl: $(NH_4)_2SO_4$ results in a good quality product (Na_2SO_4) with yield of about 95%.

Purity of the product (Na_2SO_4). The chemical composition of sodium sulphate was determined. Various batches of the product were analysed [11]. The results are shown in Table 6.

Results and Discussion

The largest use of sodium sulphate by far is the Kraft Pulp industry. It is also employed in the manufacture of window glass, in textile industry and to some extent in medicines as laxative.

The most significant economic aspect concerning the manufacture of sodium sulphate is that the supply do not appear to be in balance with the demand [2]. To encounter the growing demand of the sodium sulphate, it was considered to prepare the chemical from the naturally available rock salt (NaCl) and amonium sulphate by the foregoing process.

The study carried out indicates that sodium sulphate could readily be prepared from rock salt and ammonium sulphate at a temperature of 350° without the use of any catalyst. From the results, it is found that sodium sulphate can be achieved by the interaction of sodium chloride and ammonium sulphate by heating the mixture at 350° whereby ammonium chloride sublimes at 330° and sodium sulphate of commercial grade is obtained. The yield of this compound was investigated by studying the effect of temperature, time and stoichiometric ratios. Under the stated conditions 95% yield of sodium sulphate was achieved. The studies undertaken for the preparation of sodium sulphate is both economical and simple, as its raw material i.e. sodium chloride and ammonium sulphate are inexpensive and abundantly available within the country.

Moreover, the process involve a simple electric tube furnace where the reactants are heated at a specific tempera-

TABLE 3. EFFECT OF TEMPERATURE ON THE CONVERSION OF SODIUM CHLORIDE AND AMMONIUM SULPHATE INTO SODIUM SULPHATE.

Reactants ratio NaCl:$(NH_4)_2SO_4$	Temp. (°C)	Time (hr.)	Conversion (%)
2:1	200	1	75.3
"	250	"	80.60
"	300	"	85.40
"	350	"	95.20
"	400	"	95.2
"	450	"	95.2

TABLE 4. EFFECT OF TIME ON THE CONVERSION OF SODIUM CHLORIDE AND AMMONIUM SULPHATE INTO SODIUM SULPHATE.

Reactants ratio NaCl : $(NH_4)_2SO_4$	Temp. (°C)	Time (hr.)	Conversion (%)
2:1	350	15	80.2
"	"	30	85.3
"	"	45	90.5
"	"	60	95.5
"	"	75	95.5
"	"	90	95.5

TABLE 5. EFFECT OF REACTANTS RATIO ON THE CONVERSION OF SODIUM CHLORIDE AND AMMONIUM SULPHATE INTO SODIUM SULPHATE.

Reactants ratio NaCl:$(NH_4)_2SO_4$	Temp. (°C)	Time (hr.)	Conversion (%)
1:1	350	1	80.3
1:1 1/2	"	"	85.2
1:2	"	"	91.3
2:1	"	"	95.2
2:1 1/2	"	"	95.2
2:2	"	"	95.2

TABLE 6. COMPOSITION OF THE END PRODUCT Na_2SO_4.

	1	2	3	4	5	6
Water insoluble matter	0.005%	0.006%	0.005%	0.003%	0.004%	0.005%
Sulphur trioxide (SO'_3)	55.8%	55.3%	55.6%	55.9%	56.00%	55.7%
Sodium oxide (Na_2O)	42.7%	42.5%	42.6%	42.8%	42.8%	42.6%
Chlorides (Cl')	1.2%	1.3%	1.1%	1.2%	1.0%	1.5%
Calcium oxide (CaO)	0.002%	0.002%	0.003%	0.003%	0.002%	0.002%
Magnesium oxide (MgO)	Nil	Traces	Nil	Nil	Traces	Nil
Potassium oxide (K_2O)	Nil	Nil	Nil	Nil	Nil	Nil
Phosphorous pentoxide (P_2O_5)	Nil	Nil	Nil	Nil	Nil	Nil

Average composition of Na_2SO_4 = 98.4%.

ture for a specific time to produce sodium sulphate and therefore no complicated operational units are required for the purpose.

Conclusion

The results achieved towards the above were purely applied to R&D work which could lead to development of a process on commercial scale for this industrially important chemical. Keeping in view the growing demand of this product the studies were undertaken to prepare the chemical from the most abundantly and locally available raw materials. The production of the sodium sulphate by the process developed will save foreign exchange being spent on the import of this chemical.

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