#### 1. Introduction:

Salt, sodium chloride, has probably been with us from the beginnings of geologic time, and has been necessary directly or indirectly through all stages of evolution of living things. The first oceans undoubtedly contained traces of salt, and our unicellular ancestors first appeared and thrived in this salty marine environment.

In the process of evolution, these unicellular ancestors became multi-cellular, and some left their salty marine environment but still required salt. Our herbivorous ancestors used salt licks, and our carnivorous ones obtained their salt form the flesh and blood of their prey. This demand is necessity of/or all living for things for salt in one form or another continues today. Our bodies contain relatively large quantities of salt, as we may infer from the taste of "blood, sweat, and tears." (Dalf.w. Knutmann, 1971)

Salt, NaCl, is a chemical compound made of sodium and chloride which has been exceptionally important to humans for thousands of years, because it is one of the substances upon which all of life evolved to depend. Humans, like all life, need a supply of salt in order to simply survive. Salt's ability to preserve food was a foundation of civilization. It helped to eliminate the dependence on the seasonal availability of food and it allowed travel over long distances. However, salt was difficult to obtain, and so it was a highly valued trade item to the point of being considered a form of currency by certain peoples. Many salt roads, such as via salaria in Italy, had been established by the Bronze Age.

Today, salt is almost universally accessible, relatively cheap and often iodized.(Kurlansky, Mark., 2003).

## 1.1The Etymology of "salt":

The importance of salt in all recorded civilizations may be gauged by the number of "salt" word in the contemporary literature.

In the Medieval Latin of the middle Ages, 6<sup>th</sup> to 16<sup>th</sup> centuries widely used in Europe.

The word salt is derived from the Latin sal (=salt) and both mineral name halite and the Latin salare derived from the Greek hals. Before any knowledge, of composition of matter, sal (and later,) was applied to any sold, soluble, non inflammable substance and properties of cured sea salt thecritrion of taste was not used until early in 18<sup>th</sup>century,when began the classification acid salt, alkaline salts and neuteral salts corresponding to our modern acids alkalis and salts. Glocker in 1847 was apparently the first to use the namehlites. This was adopted in the United States by Dana in 1868 as halite, while German mineralogists used the term halit.

In English, when referring specifically to the naturally occurring substance, either the term rock salt or mineral name halite is used .In German ,common mineral terms are steinsalz, bergsalz, and halit. The French use the terms selgemme, sel de roche and selfossile for the mineral Spanish, selgema and salpiedra are used for rock salt. The Spanish mineral name in common use in Italian is alite and salgemma.(Dalf.w.Knutmann, 1971)

#### 1.2Chemistry of sodium chloride:

It is impossible to write a chronological history of the discovery of production, and early uses of salt. These probably developed as slowly as mankind itself. Salt was undoubtedly the first chemical compound (except water) which attracted the attention of man in his progress from savagery to civilization. It is the only stone eaten directly by man, and is called by Maurus Jokai "the Edible Gem." When we pick up the trail at the beginnings of recorded history, we find salt literally

everywhere. It was even then being made by solar evaporation of sea water, salt lakes, and salt springs; by quarrying and mining; by burning salt plants and eatingthe ash; by pouring brine over glowing wood coals; by direct fire evaporation of brine in pottery vessels; and by innumerable variations of such processes.

Manufacture of salt was undoubtedly initiated by discovery of its value for seasoning foods. Attempts were made by the ancient Chinese, Egyptians and Hindus to establish the origin of this salt usage but it had been used for so many centuries antedating written record that no data were even obtained. Calvert states that salt was probably used in cooking wheat and barley five thousand years ago, according to the evidence of grain varieties and pottery fragments found in Belgian cave-man dwellings. In ancient Troy, salt preserved fish were eaten some thirteen centuries before Christ Herodotus noted the Egyptians salted ducks, quail, and a sardine of the Nile.(Dalf.w.Knutmann, 1971)

#### 1.3 Salt Sources and methods of recovery:

The salt sources in approximate order of world importance today are:

- (1) Brine: (a) ocean, (b) salt lakes, (c) natural brines.
- (2) Rock salt deposits: (a) artificial well brines, (b) mines, (c) quarries.
- (3) Chemical reactions producing co-product and by product salt.
- (4)Salt –containing substances: shrubs plants, grasses.
- (5) Salt impregnated substances: peat, muds.

(Dalf.w.Knutmann, 1971)

A major change in salt production technology occurred from late Roman times (or later) when a new process was introduced in which brine was used instead of sea water. The brine was made by sand washing (also known as sleeching). Between

successive maximum (spring) high tides, there is a period of about three weeks during which the salt water impregnated ground, near the high water mark, may dry out by natural evaporation so that its surface contains dried salt. The surface sand was scraped off the beach (or other littoral site) and stored in primitive roofed enclosures. This salt-impregnated material was then washed with fresh water to produce brine and then filtered into sunken receptacles or 'sumps'.

(E.L. MorrisT. Lane and (eds.), 2001)

The methods of recovery, in tentative order of historic development are:

- (1) Solar Evaporation of brines.
- (2) Quarrying of Solid Salt.
- (3) Mining of Solid Salt.
- (4) Burning of Salt-Containing Plants.
- (5) Evaporation of brine on burning Wood.
- (6) Evaporation of brine in Vessels
- (7) Leaching of salt impregnated peat or peat ashes, muds, followed byevaporation of brine.
- (8) Sublimation of sea –water ice or sodium chloride dihydrate in cold climates.
- (9) Recovery of co product or and by product salt from chemical reactions.
- (Dalf.w.Knutmann, 1971)

## 1.3.1 Solar Evaporation of brines:

The earliest discoveries of salt may have been as an incrustation on exposed seaweeds or shore rocks, or as an efflorescence around salt springs or the salt licks of animals. The first salt production of definite record was solar salt made in Italy, although the Phoenicians must have traded in salt around the Mediterranean long before.

Pliny dwells at length on "artificial" salt; "All salt is either native or artificial; both kinds being formed in various ways, but produced from one of these two causes, the condensation or the desiccation, of liquid. Sea water, again, spontaneously produces another kind of salt, from the foam which it leaves on shore at high-water mark, or adhering to rocks; this being, in all cases, condensed by the action of the sun, and that salt being the most pungent of the tow which is found upon the rocks."

Pliny continues: "Of artificial salt there are several kinds; the common salt, the most abundant, being made from Sea -water drained into salt -pans, ... On the shores of Egypt, salt is formed by overflow of sea upon the land, already prepared for its reception, ... It is made here, also, by the waters of certain wells discharged into salt -pans ..."

In solar evaporation, the salt crystals formed on the brine surface and a held there by surface tension until too large to float or until disturbed they have a characteristic flaky or ribbed hopper shape, and are often found broken into fragments.(Dalf.w.Knutmann, 1971)

## 1.3.2Quarrying of Solid Salt:

In many parts of arid North Africa both rock salt and stone were abundant, but since the salt was easier to work, blocks of salt cemented with mud, were used for building houses. Near Timbuktu, house walls of salt were roofed with camel skins. The salt was found in clearly marked layers under a thin layer of sand, and wastrimmed into slabs approximately 3ft 6in.by 1 ft 3., in appearance like bars of red or gray veined marble.(Dalf.w.Knutmann, 1971)

## 1.3.3Mining of Solid Salt:

Rock salt was known and used by ancients ceremonials, arts, and a flavor and preservative, although its chemical identity with sea salt was not known until the 18<sup>th</sup> century. The first rock salt used was probably quarried from exposed deposits in arid regions, and this would logically be followed by lateral digging into the sides the quarries, thus leading to true mining.

The rock salt mines of the eastern Alps were operated from the later Bronze Age, about 1400B.C.and possibly earlier. Rock salt deposits in the foothills of Sierra Nevada in Spain had been mined long before the 5<sup>th</sup> century B.C. The salt mines of Northern India were worked before the time of Alexander, 356 to 323 B.C. The mining of rock salt on an extensive scale was started about A.D.1000 at the world –famous wiczka mine in Galicia, Poland, and has been conducted up to the present.(Dalf.w.Knutmann, 1971)

## 1.3.4Burning of Salt-Containing Plants:

Many savage tribes far inland from the solar salt deposits of the sea have apparently know of the saline taste of certain plant ashes, and obtained a salt equivalent thus. Certain New Guinea natives obtain salt by an elaborate method of leaching plant ashes and evaporating the brine.

The Virginia and other Indians used saline ashes of certain plants such as the saltwort as a substitute for salt. The plant known as colts-foot was also used by the Indians. They rolled the green leaves into small walls; carefully sun dried them, and burned them to a very saline ash on a flat rock. The Indians in the areas of the first settlements knew nothing of salt as such, and used hickory and stickweed ashes.(Dalf.w.Knutmann, 1971)

## 1.3.5 Evaporation of brine on burning Wood:

In areas where the solar method was unknown, or the climate unsuitable, another method was used; probably the earliest nonsolar method.(Dalf.w.Knutmann, 1971)

Wood was initially the fuel used in the evaporation process, but sources of wood became scarce before long due to the scale of the salt production industry. Several energy saving techniques were used during evaporation, but natural gas eventually replaced wood in the brine evaporation process. At some point in the 16th century, techniques were developed to harness the natural gas encountered during drilling for brine. Natural gas was burned beneath the big salt pans. The introduction of natural gas and its coexistence with brine pushed Zigong's salt production into the industrial scale. (MarkKurlansky, 2004)

#### 1.3.6Evaporation of brine in Vessels:

Probably the earliest artificial evaporated salt was made by dropping brine on burning wood coals or heated rocks and scraping off the efflorescence. This method was used in Europe and in England until Caesear's salinators taught the natives of Cheshire boil the brine in small shallow lead evaporating pans over open fires. During medieval times the lead pans replaced by iron and then by steel while coal the wood fuel. For 1700 years all Cheshire salt was made in this way, although with introduction of increasingly larger pans.

The Chinese had undoubtedly known of methods of preparing a superior quality of salt from brine long before its first mention in the annals of the Emperor Yu, 2205 to 2197BC, when he ordered the province of Shantung to supply the court with salt.

The modern vacuum pan method of producing salt in fine cubic crystal was first used in 1886, so all pervious artificial production methods were necessarily of the open-vessel type, which produced flakes; cubes or mixture of the two. Quiet surface evaporation produce flakes; turbule boiling, cubes.(Dalf.w.Knutmann, 1971)

# 1.3.7Leaching of peat Impregnated with Salt:

In Holland, long before the Carolingian period and up to the 15<sup>th</sup> century, sea water –impregnated peat was burned, the ashes leached to produce strong brine, and the latter evaporated in small, light pans over turf fires.

(Dalf.w.Knutmann, 1971)

#### 1.3.8 Sublimation in cold climates:

Sea water ice contains substantial quantities of salt. Further, production of ice from sea water, salt lakes an springs results in concentration of remaining brine, and production of substantial quantities of sodium chloride dihydrate. Sublimation of the Sea water ice, and melting or sublimation of dihydrate the results in crystalline salt. These accidental production methods have been used in very cold climates since antiquity. (Dalf.w.Knutmann, 1971)

## 1.3.9 Production of Co - Production or By - Product Salt:

The largest source of by –product salt is during the concentration of calcium chloride brines resulting from soda ash manufacture. Salt is also produced on large scale by use sodium to reduce metallic chlorides such as TiCl<sub>4</sub>. It is also a byproduct in the manufacture of various organic and inorganic chemical all these production methods are comparatively recent, tied in with the growth of chemical. (Dalf.w.Knutmann, 1971)

#### 1.4Sodium chloride:

Sodium chloride common salt, NaCl salt is found in sea water, in salt wells, Inland (eg .sambhar in India and Lake Elton in Russia) and In deposits of rock salt in Himachal Pradesh (Mandi) and Khewra (Punjab,Pakistan) Rock salt is dug out or dissolved in water if found very deep .The saturated solution is pumped out and evaporated to get the salt. It is also manufactured from sea water and water of certain lakes by evaporation by Solar heat and wind when sodium chloride separates out .Sea water is run in to prevent percolation and allowed to evaporate .clay deposits here and the saturated solution is made to flow into other lagoons and evaporates further when crude salt is deposited and is raked up. The mother – liquor known as bittern may be used for manufacture of magnesium and bromine.

In cold countries, like Russia, sea water is taken Into pits where only water freezes night leaving a concentrated solution. The percentage rises daily till it is 22 percent and about 90 percent of water has been removed. It is colourless crystalline substance (cubic crystals, m.p.1093k) solubility does not vary appreciably with rise or fall of temperature (273 to252k) a dehydrate . NaCl.2H<sub>2</sub>O exists.

On heating, the crystal break up with a crackling noise .Ordinary salt is slightly hygroscopic due to trace of magnesium and calcium chloride present .It gives the common reactions of a chloride and soluble sodium ions.

U se. (I) it is an essential constituent of our diet.

- (ii) As preservative, e.g. in packing and curing of meat and fish.
- (iii) Mixed with ice, it gives a freezing mixture.
- (iv) As starting material for the manufacture of chlorine, hydrochloric acid, washing soda, caustic soda, and many other sodium compounds.
- (v) In the preparation of pottery glaze.
- (iv) In the manufacture of soap, for salting out. (P.l.son1and Mohan,1991)

Table 1: some properties of sodium chloride

Sodium chloride	
IUPAC name	
Sodium chloride	
Other names	
Common salt	
Halite	
Rock salt	
Saline	
Sodium chloric	
Table salt	
Properties	
Formula	NaCl
Molar mass	58.44 g mol <sup>-1</sup>
Appearance	Colorless crystals
Odor	Odorless
Density	2.165 g/cm <sup>3</sup>

Melting point 801 °C

Boiling point 1,413 °C

Solubility in water 359 g/L

Solubility in ammonia 21.5 g/L

Solubility in methanol 14.9 g/L

Refractive index  $(n_D)$  1.5442 (at 589 nm)

Structure

Crystal structure Cubic

Space group Fm3m, No. 225

Lattice constant a = 564.02 pm

Coordination

geometry Octahedral (Na<sup>+</sup>)

Octahedral (Cl<sup>-</sup>)

Thermochemistry

Specific  $36.79 \text{ J K}^{-1} \text{ mol}^{-1}$ 

heat capacity (C)

Std  $\operatorname{molar} 72.11 \ \operatorname{J} \ \operatorname{K}^{-1} \ \operatorname{mol}^{-1}$ 

entropy  $(S_{298}^{\Theta})$ 

Stdenthalpyof formation ( $\Delta_f H^{\Theta}_{298}$ )  $-411.12 \text{ kJ mol}^{-1}$ 

Sodium chloride, (Wells,JohnC.,2008) also known as salt, common salt, table salt or halite, is an ionic compound with the chemical formulaNaCl, representing a 1:1 ratio of sodium and chlorine atoms. Sodium chloride is the salt most responsible for the salinity of the seawater and of the extracellular fluid of many multicellular organisms. In the form of edible or table salt it is commonly used as a condiment and food preservative. Large quantities of sodium chloride are used in many industrial processes, and it is a major source of sodium and chlorine compounds used as feedstocks for further chemical syntheses. A second major consumer of sodium chloride is de-icing of roadways in sub-freezing weather.

#### 1.4.1 Solid sodium chloride:

In solid sodium chloride, each ion is surrounded by six ions of the opposite charge as expected on electrostatic grounds. The surrounding ions are located at the vertices of a regular octahedron. In the language of close-packing, the larger chlorideions are arranged in a cubic array whereas the smaller sodium ions fill all the cubic gaps (octahedral voids) between them. This same basic structure is found in many other compounds and is commonly known as the halite or rock-salt crystal structure. It can be represented as a face-centered cubic (fcc) lattice with a two-atom basis or as two interpenetrating face centered cubic lattices. The first atom is located at each lattice point, and the second atom is located half way between lattice points along the fcc unit cell edge.

Thermal conductivity of NaCl as a function of temperature has a maximum of 2.03 W/(cm K) at 8 K (-265.15 °C; -445.27 °F) and decreases to 0.069 at 314 K (41 °C; 106 °F). It also decreases with doping. (Sirdeshmukh, Dinker B.; Sirdeshmukh, Lalitha and Subhadra, K. G. ,2001)

# 1.4.2 Aqueous solution:

The attraction between the Na<sup>+</sup> and Cl<sup>-</sup> ions in the solid is so strong that only highly polar solvents like water dissolve NaCl well.

Table2: Solubility of NaCl in various solvents

Solubility	of	NaCl	in	various	solvents
(g NaCl / 1 k	g of sol	vent at 25	°C (77	° <b>F</b> ))  (Burgess	s, J. ,1978)
Water				360	
Formamide				94	
Glycerin				83	
Propylene gly	ycol			71	
Formic acid				52	
Liquid ammo	nia			30.2	
Methanol				14	
Ethanol				0.65	
Dimethylforn	namide			0.4	
1-Propanol				0.124	
Sulfolane				0.05	
1-Butanol				0.05	
2-Propanol				0.03	
1-Pentanol				0.018	
Acetonitrile				0.003	
Acetone				0.00042	ļ

When dissolved in water, the sodium chloride framework disintegrates as the Na<sup>+</sup> and Cl<sup>-</sup> ions become surrounded by the polar water molecules. These solutions consist of metal aquo complex with the formula [Na(H<sub>2</sub>O)<sub>8</sub>]<sup>+</sup>, with the Na-O distance of 250 pm. The chloride ions are also strongly solvated, each being surrounded by an average of 6 molecules of water. (Lincoln, S. F.; Richens, D. T. and Sykes, A. G., 2003) Solutions of sodium chloride have very different properties from pure water. The freezing point is –21.12 °C (–6.02 °F) for 23.31 wt% of salt, and the boiling point of saturated salt solution is near 108.7 °C (227.7 °F). (Elvers, B. *et al.* (ed.), 1991 )

From cold solutions, salt crystallises as the dihydrate NaCl·2H<sub>2</sub>O.

# **1.4.3** Unexpected stable stoichiometric variants:

Common salt has a well-established 1:1 molar ratio of sodium and chlorine. In 2013, compounds of sodium and chloride of different stoichiometries have been discovered by a team at Stony Brook University; five new compounds were predicted (e.g., Na<sub>3</sub>Cl, Na<sub>2</sub>Cl, Na<sub>2</sub>Cl, Na<sub>2</sub>Cl<sub>2</sub>, NaCl<sub>3</sub>, and NaCl<sub>7</sub>). The existence of some of them has been tested and experimentally confirmed: cubic and orthorhombic NaCl<sub>3</sub> and two-dimensional metallic tetragonal Na<sub>3</sub>Cl. This indicates that compounds violating chemical intuition are possible, in simple systems under nonambient conditions. ( Zhang, Oganov,Goncharov, Zhu, Boulfelfel, Lyakhov, Stavrou, Somayazulu, Prakapenka, Konôpková, , 2013).

#### 1.4.4 Occurrence:

Small particles of sea salt are the dominant cloud condensation nuclei well out at sea, which allow the formation of clouds in otherwise non-pollutedair. (Mason, 2006).

#### 1.4.5 Production:

Salt is currently mass-produced by evaporation of seawater or brine from brine wells and salt lakes. Mining of rock salt is also a major source. China is the world's main supplier of salt (Dennis S. Kostick, 2008) In 2010, world production was estimated at 270 million tonnes, the top five producers (in million tonnes) being China (60.0), United States (45.0), Germany (16.5), India (15.8) and Canada (14.0). (Salt, U.S. Geological Survey,2001)Salt is also a byproduct of potassium mining.

#### 1.4.6 Uses:

In addition to the familiar domestic uses of salt, more dominant applications of the approximately 250 megatons/year production (2008 data) include chemicals and de-icing. (Westphal, Gisbert*et al.*, 2002)

### 1.4.7 Chemicals production:

Salt is the source, directly or indirectly, for the production of many chemicals, which consume most of the world's production. (Dennis S. Kostick, 2008)

## 1.4.8 Chlor-alkali industry:

It is the starting point for the Chloralkali process, which provides the world with chlorine and sodium hydroxide according to the chemical equation

$$2 \ NaCl + 2 \ H_2O \rightarrow Cl_2 + H_2 + 2 \ NaOH$$

This electrolysis is conducted in either a mercury cell, a diaphragm cell, or a membrane cell. Each of those use a different method to separate the chlorine from the sodium hydroxide. Other technologies are under development due to the high energy consumption of the electrolysis, whereby small improvements in the efficiency can have large economic paybacks. Some applications of chlorine include PVC, disinfectants, and solvents. Sodium hydroxide enables industries that produce paper, soap, and aluminium.

## 1.4.9 Soda ash industry:

Via the Solvay process, sodium chloride is used to produce sodium carbonate and calcium chloride. Sodium carbonate is used to produce glass, sodium bicarbonate, and dyes as well as a myriad of other chemicals.

Solvay process

$$NaCl + NH_4HCO_3 \rightarrow NaHCO_3 + NH_4Cl$$
 .....(1)

$$2NaHCO_3+\Delta \rightarrow Na_2CO_3+CO_2+H_2O$$
 .....(2)

$$2NaCl + CaCO_3 \rightarrow Na_2CO_3 + CaCl_2...$$
 (3)

(Dalf.w.Knutmann, 1971)

In the Mannheim processand in the Hargreaves process, it is used for the production of sodium sulfate and hydrochloric acid.

Mannheim process

$$(2)$$
NaCl+NaHSO<sub>4</sub> $\rightarrow$ Na<sub>2</sub>SO<sub>4</sub>+HCl

Hargreaves process

$$4NaCl+2 SO_2+2H_2O+O_2 \rightarrow 2Na_2SO_4+4HCl$$

(Dalf.w.Knutmann, 1971)

#### **1.4.10 Standard:**

Sodium chloride has an international standard that is created by ASTM International. These methods listed provide procedures for analyzing sodium chloride to determine if it is suitable for its intended use and application.

#### 1.4.11 Miscellaneous industrial uses:

Sodium chloride is heavily used, so even miscellaneous applications can consume massive quantities. In oil and gas exploration, salt is an important component of drilling fluids in well drilling. It is used to flocculate and increase the density of the drilling fluid to overcome high downwell gas pressures. Whenever a drill hits a salt

formation, salt is added to the drilling fluid to saturate the solution and to minimize the dissolution within the salt stratum. (Westphal, Gisbert*et al.*, 2002)

Salt is also used to increase the curing of concrete in cemented casings. (Dennis S. Kostick, 2008)

In textiles and dyeing, salt is used as a brine rinse to separate organic contaminants, to promote "salting out" of dyestuff precipitates, and to blend with concentrated dyes to standardize them. One of its main roles is to provide the positive ion charge to promote the absorption of negatively charged ions of dyes. (Dennis S. Kostick, 2008)

It also is used in processing aluminium, beryllium, copper, steel and vanadium. In the pulp and paper industry, salt is used to bleach wood pulp. It is also used to make sodium chlorate, which is added along with sulfuric acid and water to manufacture chlorine dioxide, an excellent oxygen-based bleaching chemical. The chlorine dioxide process, which originated in Germany after World War I, is becoming more popular because of environmental pressures to reduce or eliminate chlorinated bleaching compounds. In tanning and leather treatment, salt is added to animal hides to inhibit microbial activity on the underside of the hides and to attract moisture back into the hides. (Dennis S. Kostick, 2008).

In rubber manufacture, salt is used to make buna, neoprene and white rubber types. Salt brine and sulfuric acid are used to coagulate an emulsified latex made from chlorinated butadiene. Dennis .(SKostick, 2008) (Westphal, Gisbert*et al.*, 2002) Salt also is added to secure the soil and to provide firmness to the foundation on which highways are built. The salt acts to minimize the effects of shifting caused in the subsurface by changes in humidity and traffic load.( Dennis S. Kostick, 2008)

#### 1.412 Water softening:

Hard water contains calcium and magnesium ions that interfere with action of soap and contribute to the buildup of a scale or film of alkaline mineral deposits in household and industrial equipment and pipes. Commercial and residential water-softening units use ion exchange resins to remove the offending ions that cause the hardness. These resins are generated and regenerated using sodium chloride. (Westphal, Gisbert*et al.*, 2002)

#### **1.4.13 Road salt:**

The second major application of salt is for de-icing and anti-icing of roads, both in grit bins and spread by winter service vehicles. In anticipation of snowfall, roads are optimally "anti-iced" with brine (concentrated solution of salt in water), whichprevents bonding between the snow-ice and the road surface. This procedure obviates the heavy use of salt after the snowfall. For de-icing, mixtures of brine and salt are used, sometimes with additional agents such as calcium and magnesium chloride. The use of salt or brine becomes ineffective below  $-10~^{\circ}\text{C}$  (14  $^{\circ}\text{F}$ ).

Salt for de-icing in the United Kingdom predominantly comes from a single mine in Winsford in Cheshire. Prior to distribution it is mixed with <100 ppm of sodium hexacyanoferrate(II) as an anti-caking agent, which enables rock salt to flow freely out of the gritting vehicles despite being stockpiled prior to use. In recent years this additive has also been used in table salt. Other additives had been used in road salt to reduce the total costs. For example, in the US, a byproduct carbohydrate solution from sugar beet processing was mixed with rock salt and adhered to road surfaces about 40% better than loose rock salt alone. Because it stayed on the road longer, the treatment did not have to be repeated several times, saving time and money. (Dennis S. Kostick, 2008)

In the technical terms of physical chemistry, the minimum freezing point of a water-salt mixture is -21.12 °C (-6.02 °F) for 23.31 wt% of salt. Freezing near

this concentration is however so slow that the eutectic point of -22.4 °C (-8.3 °F) can be reached with about 25 wt% of salt.(Elvers, B. *et al.* (ed.), 1991)

#### 1.4.14 Environmental effects:

Road salt ends up in fresh water bodies and could harm aquatic plants and animals by disrupting their osmoregulation ability.(Rastogi, 2010) The omnipresence of salt poses a problem in any coastal coating application, as trapped salts cause great problems in adhesion. Naval authorities and ship builders monitor the salt concentrations on surfaces during construction. Maximum salt concentrations on surfaces are dependent on the authority and application. The IMO regulation is mostly used and sets salt levels to a maximum of 50 mg/m² soluble salts measured as sodium chloride. These measurements are done by means of a Bresle test.

In highway de-icing, salt has been associated with corrosion of bridge decks, motor vehicles, reinforcement bar and wire, and unprotected steel structures used in road construction. Surface runoff, vehicle spraying, and windblown actions also affect soil, roadside vegetation, and local surface water and groundwater supplies. Although evidence of environmental loading of salt has been found during peak usage, the spring rains and thaws usually dilute the concentrations of sodium in the area where salt was applied. (Dennis S. Kostick, 2008)

#### 1.4.15 Food industry, medicine and agriculture :

Many micro organisms cannot live in an overly salty environment: water is drawn out of their cells by osmosis. For this reason salt is used to preserve some foods, such as smoked bacon, fish, or cabbage. It can also be used to detach leeches that have attached themselves to feed.

Salt is added to food, either by the food producer or by the consumer, as a flavor enhancer, preservative, binder, fermentation-control additive, texture-control agent and color developer. The salt consumption in the food industry is subdivided, in descending order of consumption, into other food processing, meat packers,

canning, baking, dairy and grain mill products. Salt is added to promote color development in bacon, ham and other processed meat products. As a preservative, salt inhibits the growth of bacteria. Salt acts as a binder in sausages to form a binding gel made up of meat, fat, and moisture. Salt also acts as a flavor enhancer and as a tenderizer. (Dennis S. KostickSalt, , 2008)

In many dairy industries, salt is added to cheese as a color-, fermentation-, and texture-control agent. The dairy subsector includes companies that manufacture creamery butter, condensed and evaporated milk, frozen desserts, ice cream, natural and processed cheese, and specialty dairy products. In canning, salt is primarily added as a flavor enhancer and preservative. It also is used as a carrier for other ingredients, dehydrating agent, enzyme inhibitor and tenderizer. In baking, salt is added to control the rate of fermentation in bread dough. It is also used to strengthen the gluten (the elastic protein-water complex in certain doughs) and as a flavor enhancer, such as a topping on baked goods. The food-processing category also contains grain mill products. These products consist of milling flour and rice and manufacturing cereal breakfast food and blended or prepared flour. Salt is also used a seasoning agent, e.g. in potato chips, pretzels, cat and dog food. (Dennis S. KostickSalt, 2008)

Sodium chloride is used in veterinary medicine as emesis causing agent. It is given as warm saturated solution. Emesis can also be caused by pharyngeal placement of small amount of plain salt or salt crystals.

## 1.4.16 Firefighting:

Sodium chloride is the principal extinguishing agent in fire extinguishers (Met-L-X, Super D) used on combustible metal fires such as magnesium, potassium, sodium, and NaK alloys (Class D). Thermoplastic powder is added to the mixture,

along with waterproofing (metal stearates) and anti-caking materials (tricalcium phosphate) to form the extinguishing agent. When it is applied to the fire, the salt acts like a heat sink, dissipating heat from the fire, and also forms an oxygen-excluding crust to smother the fire. The plastic additive melts and helps the crust maintain its integrity until the burning metal cools below its ignition temperature. This type of extinguisher was invented in the late 1940s in the cartridge-operated, although stored pressure versions are now popular. Common sizes are 30 pounds (14 kg) portable and 350 pounds (160 kg) wheeled

#### **1.4.17 Cleanser:**

Since at least medieval times, people have used salt as a cleansing agent rubbed on household surfaces. It is also used in many brands of shampoo, toothpaste and popularly to de-ice driveways and patches of ice.

#### 1.4.18 Optical usage:

Defect-free NaCl crystals have an optical transmittance of about 90% between 200 nm and 20  $\mu$ m. They were therefore used in optical components (windows and prisms) operating in the infrared spectral range, where few non-absorbing alternatives exist and where requirements for absence of microscopic inhomogeneities are less strict than in the visible range. While inexpensive, NaCl crystals are soft and hygroscopic – when exposed to the ambient air they gradually cover with "frost". This limits application of NaCl to dry environments or for short-term uses such as prototyping. Nowadays materials like zinc selenide (ZnSe), which are stronger mechanically and are less sensitive to moisture, are used instead of NaCl for the IR spectral range.

## 1.4.19 Biological functions:

The long held belief that a high-salt diet raises the risk of cardio-vascular disease is coming under scrutiny. (McCarron, David, 2008) More recently, dietary salt was

demonstrated to attenuate nitric oxide production. Nitric oxide (NO) contributes to vessel homeostasis by inhibiting vascular smooth muscle contraction and growth, platelet aggregation, and leukocyte adhesion to the endothelium. (Osanai, Fujiwara, Saitoh, *et al.*, 2002)

#### 1.5 Manufacture:

Salt occurs throughout the United States. In 1972, Louisiana was the salt-producing state, with 30% of total output; Texas was second, with 21%. New York and Ohio each produced about 13%, Michigan about 10%, and California, Kansas, and West Virginia each 3%.

Salt is obtained in three different ways namely, solar evaporation of sea water on pacific coast or from western salt-lake brines, from mining of rock salt, and from well brines.

The purity of the salt obtained from the evaporation of salt water is usually about 98% to 99%. Mined salt varies very widely in composition, depending on the locality. Some rock salt, however, runs as 99.5% pure. The solution obtained from wells is usually about 98% pure and depends to a great extent on the purity of the water forced down into the well to dissolve the salt from the rock bed. For many purposes, the salt obtained from mines and by direct evaporation of salt solutions is pure enough for use; however, a large portion must be purified to remove such materials as calcium and magnesium chlorides.

Solar evaporation is used extensively in dry climates, where the rate of evaporation depends on the humidity of air, wind velocity, and amount of solar energy absorbed. San Francisco, southern California, and the Great Salt Lake area are the primary producing areas in the United States. Solar evaporation represents only a small part of the U.S.production but a large part of the world production.

The mining of rock salt uses methods similar to coal mining. As the salt is removed, large rooms are formed, supported by pillars of salt.

Salt brine is obtained by pumping water into a salt deposit and bringing the salt to the surface for further purification.

In shallow wells a double-pipe system of pumping water down one pipe and recovered brine up the other has been used for years. However, in newer wells a series of wells is drilled to the base of the salt cavity, water is injected under pressure into one well. And the brine is removed from a nearby well when the two are interconnected by dissolving the salt between the two wells. This known as hydraulic fracturing and has reduced the time required to achieve maximum production.

The vacuum pan system, employing multiple-effect evaporators, is the most common method of producing salt from brine. The type of evaporator may vary, but modern units are generally forced-circulation units constructed of monel. Evaporation processes are also under development. (Shreve and Brink, 1969).

# 1.5.1From saturated brine by multiple-effect a process:

Saturated brine for the production of "evaporated" salt is usually obtained by pumping water into an underground salt deposit and removing the saturated solution from an adjacent interconnected well, or from the same well by means of an annular pipe. Besides sodium chloride, the brine will contain more or less calcium sulfate, calcium chloride, and magnesium chloride and ferrous ions plus traces of hydrogen sulphide and ferrous ions (Fig.1). Such a solution may have the following approximate composition:

Other minor impurities account for the remainder. Dissolved hydrogen sulphide  $(H_2S)$  may amount to about 0.015%.

Table2:Ingredients and Percent From saturated brine by multiple-effect evaporation process.

Water	73.5
Sodium chloride	26.3
Calcium sulphate	0.12
Calcium chloride	0.003
Magnesium	0.007
chloride	
	99.93

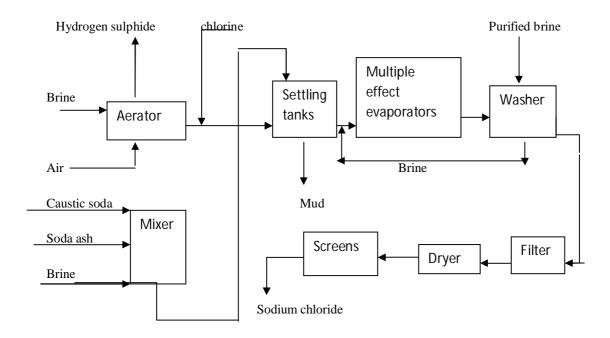


Fig.1. Flow diagram for manufacture of sodium chloride from saturated brine by multiple- effect evaporation.

In the chemical treatment, the brine receives varies from one plant to another, and in a few cases, where very pure rock salt deposits are available; it may require none at all. Typically, however, the brine is first aerated to remove most of the hydrogen sulphide. Addition of a small amount of chlorine will complete hydrogen sulphide removal by oxidation, and also oxidize ferrous ions to ferric. The brine is then pumped to settling tanks, where it may be treated with a dilute solution of

caustic soda and soda ash to remove most of the calcium, magnesium, and ferric ions. After clarification the treated brine is pumped to multiple-effect evaporators. As water is removed, salt crystals are removed as a slurry. After screening to remove lumps, the slurry is sent to a conical washer, where the salt crystals are washed counter-currently with fresh brine. By this washing, actually hydraulic classification, fine light crystals of calcium sulphate are removed from the mother liquor of the slurry and returned to the evaporator. Eventually, the calcium sulphateconcentration in the evaporation system builds up to the point where it must be removed by "boiling-out" the evaporators.

The washed slurry is filtered, the mother liquor is returned to the evaporators, and the salt crystals from the filter are dried and screened. Salt thus produced from the typical brine described is of 99.8% purity or greater. Many salt manufacturers do not treat the raw brine, but control calcium and magnesium impurities by watching the concentrations in evaporators and bleeding off sufficient brine to maintain a predetermined level .By such methods, salt of better than 99.5% purity can by made consistently. In either case, the final screening of the dried salt yields various grades, depending on particle size. The coarsest is industrial salt. This calcium silicate or tricalcium phosphate with the salt. Iodised salt after blending contains 0.01% potassium iodide, a stabilizer such as 0.1% sodium carbonate, 0.1% sodium thiosulphate.

# 1.5.2 From saturated brine by open (Grainer) process:

Salt in the form of a hopper-like crystal (grainer salt) is made by causing the salt crystal to form on the surface of hot, quiescent brine held in an open pan or grainer. The grainer is a flat, open pan , 4.5 to 6 m wide, 45 to 60 m long, and about 60 cm deep. Beneath the submerged steam coils is a system of reciprocating rakes for salt removal (Fig.2)

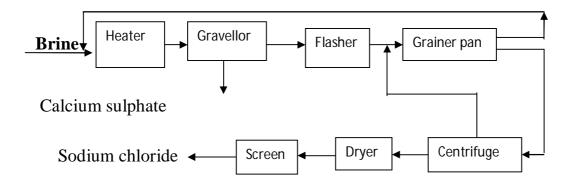


Fig.2. Flow diagram for manufacture of sodium chloride from saturated brine by open pan-Grainer process.

As in the case of vacuum-pan salt, the brine may or may not be chemically treated. Also, different methods may be used to reduce calcium sulphate contamination of the finished salt.

In the process shown in the flow diagram, saturated brine mixed with recirculating brine from the grainer is heated to 110°C, When a calcium sulphate is much less soluble than at lower temperatures. The precipitated calcium sulphate is then removed in a graveller or bed of stones. The purified brine is then flash-cooled to such a temperature that sodium chloride is still retained in solution, but any remaining calcium sulphate crystals re-dissolve. The slurry is then pumped to the grainer, where evaporation takes place slowly at 96°C. Flat, hopper-shaped crystals formed on the surface and then fall to the bottom of the grainer, where they grow further before being removed by the rake system. The wet crystals are centrifuged, dried, and screened. When the incoming brine has been treated, salt of 99.8% purity is not unusual.

Because of its high surface-volume relationship, grainer process is very wasteful of heat, and there are only a few plants operating to satisfy the demand for this special type of salt.

#### 1.5.3 From rock salt by mining:

About35% of the salt produced comes from mines. The salt deposits vary in colour from light reddish-brown to a sort of off-gray. Purity is usually about 98.5%. After chunks of salt are blasted loose, they are preliminarily crushed in the mine and then crushed again at the surface. The remaining processing consists of a series of grinding and screening operations to produce salt crystals of various sizes. The product is cheaper than evaporated salt and sold for those uses in which small amounts of impurities are allowable such as ice cream manufacture and the salting of hides. Some mines have as much as 20% waste in the form of fines. An evaporation process called the "recrystallization process" has been developed to recover these fines as high-purity evaporated salt without resorting to chemical treatment.

Calcium sulphate ting to chemical treatment. Slurry of rock salt fines is heated and sent to a saturator, where all the salt dissolves and part of the calciumsulphate separates. Final separation of salt and the remaining calcium sulphate is accomplished in a double-effect crystallizing evaporator. Salt of 99.99% purity is produced.

#### 1.5.4 From sea water by solar evaporation:

At point along the West Coast of the Great Salt Lake, salt is produced from sea water by solar evaporation. In these areas. There are no rock salt deposits, and annual evaporation exceeds precipitation. For example, during the 7-month period from April to October, there is less than 125 mm of rain, but evaporation is equivalent to about 840 mm. Here sea water (containing about 3,7% solids of which about 78% is sodium chloride) is caught in ponds at high tide. After the water evaporates to about sp.gr.1.21, at which concentration most of the calcium sulphate precipitates, it is pumped to another pond from which it flows to the rest of the system by gravity. When salt crystals start to form, the brine is run into crystallizing ponds. Here about 75% of the salt separates as the evaporation

continues. When the specific gravity reaches 1.25to 1.29, the mother liquor (bittern) contains 300to 400 g / liter of solids and is run off. It may either be discarded or worked up for other soluble salts.

At the end of the season, the ponds are drained, and the salt is harvested mechanically. It is crushed, washed with salt brine, and dried to produce an industrial grade (about 95% pure). If table salt or dairy salt is desired, the solar salt is re-dissolved and evaporated.

# 1.5.5 Other processes:

A process has been proposed whereby salt will be produced as a by- product of fresh water – by salt-water desalination. Multiple-effect evaporation of sea water would convert 75% of the water to fresh, potable water. The remaining brine would be sent to solar evaporation ponds, and the harvested salt would be purified by recrystallisation process previously described for upgrading rock salt. A similar effect would occur if other means for desalination, such as reverse osmosis or dialysis, are employed.(K.H.Davais /F.S.Berner,2004)

#### 1.6 Recrystallization technique:

#### 1.6.1 Proper purification crystalline solids:

The most common method of purifying solid organic compounds is by recrystallization. In this technique, an impure solid compound is dissolved in a solvent and then allowed to slowly crystallize out as the solution cools. As the compound crystallizes from the solution, the molecules of the other compounds dissolved in solution are excluded from the growing crystal lattice, giving a pure solid.

Crystallization of a solid is not the same as precipitation of a solid. In crystallization, there is a slow, selective formation of the crystal framework resulting in a pure compound. In precipitation, there is a rapid formation of

a solid from a solution that usually produces an amorphous solid containing many trapped impurities within the solid's crystal framework. For this reason, experimental procedures that produce a solid product by precipitation always include a final recrystallization step to give the pure compound.

## 1.6.2 The steps in the Recrystallization a compound:

Recrystallization relies on the property that for most compounds, as the temperature of a solvent increases, the solubility of the compound in that solvent also increases. For example, much more table sugar can be dissolved in very hot water (just below the boiling point) than in water at room temperature. What will happen if a concentrated solution of hot water and sugar is allowed to cool to room temperature? As the temperature of the solution decreases, the solubility of the sugar in the water also decreases and the sugar molecules will begin to crystallize out of the solution. (This is how rock candy is made.) This is the basic process that goes on in the recrystallization of a solid. ARE:

- 1. Find a suitable solvent for the recrystallization;
- 2. Dissolve the impure solid in a minimum volume of hot solvent;
- **3.** Remove any insoluble impurities by filtration;
- **4.** Slowly cool the hot solution to crystallize the desired compound from the solution;
- **5.** Filter the solution to isolate the purified solid compound.

#### 1.6.3 Choosing a solvent:

The first consideration in purifying a solid by recrystallization is to find a suitable solvent. There are four important properties that you should look for in a good solvent for recrystallization.

1. The compound should be very soluble at the boiling point of the solvent and only sparingly soluble in the solvent at room temperature. This

difference in solubility at hot versus cold temperatures is essential for the recrystallization process. If the compound is insoluble in the chosen solvent at high temperatures, then it will not dissolve. If the compound is very soluble in the solvent at room temperature, then getting the compound to crystallize in pure form from solution is difficult. For example, water is an excellent solvent for the recrystallization of benzoic acid. At 10°C only 2.1 g of benzoic acid dissolves in 1 liter of water, while at 95 °C the solubility is 68 g/L.

- 2. The unwanted impurities should be either very soluble in the solvent at room temperature or insoluble in the hot solvent. This way, after the impure solid is dissolved in the hot solvent, any undissolved impurities can be removed by filtration. After the solution cools and the desired compound crystallizes out, any remaining soluble impurities will remain dissolved in the solvent.
- **3.** The solvent should not react with the compound being purified. The desired compound may be lost during recrystallization if the solvent reacts with the compound.
- **4.** The solvent should be volatile enough to be easily removed the solvent after the compound has crystallized. This allows for easy and rapid drying of the solid compound after it has been isolated from the solution.

Finding a solvent with the desired properties is a search done by trial and error. First, test the solubility of tiny samples of the compound in test tubes with a variety of different solvents (water, ethanol, methanol, ethyl acetate, diethyl ether, hexane, toluene, etc.) at room temperature. If the compound dissolves in the solvent at room temperature, then that solvent is unsuitable for recrystallization. If the compound is insoluble in the solvent at room temperature, then the mixture is heated to the solvent's boiling point to

determine if the solid will dissolve at high temperature, and then cooled to see whether it crystallizes from the solution at room temperature.

#### 1.6.4 Dissolving the solid:

Once a suitable solvent is selected, place the impure solid in an Erlenmeyer flask and add a small volume of hot solvent to the flask. Erlenmeyer flasks are preferred over beakers for recrystallization because the conical shape of an Erlenmeyer flask decreases the amount of solvent lost to evaporation during heating, prevents the formation of a crust around the sides of the glass, and makes it easier to swirl the hot solution while dissolving the solid without splashing it out of the flask.

Keep the solution in the Erlenmeyer flask warm on a hot plate or in a water bath, and add small volumes of hot solvent to the flask until all of the solid just dissolves. Swirl the solution between additions of solvent and break up any lumps with a stirring rod or spatula. Occasionally there will be impurities present in the solid that are insoluble in the chosen solvent even at high temperature. If subsequent additions of solvent to the solution do not seem to dissolve any of the remaining solid, stop adding solvent to the solution (as this will decrease the percent recovery of the desired compound) and filter or decant the hot solution to remove the insoluble impurities.

#### 1.6.5 Using decolorizing carbon:

Colored impurities are sometimes difficult to remove from solid mixtures. These colored impurities, often due to the presence of polar or polymeric compounds, can cause a colorless organic solid to have a tint of color even after recrystallization. Decolorizing or activated carbon is used to remove the colored impurities from the sample. Decolorizing carbon isvery finely divided carbon that provides high surface area to adsorb the colored impurities.

Very little decolorizing carbon is needed to remove the colored impurities from a solution. You must be judicious in your use of decolorizing carbon: if too much is used, it can adsorb the desired compound from the solution as well as the colored impurities. After the impure solid sample is dissolved in hot solvent, a small amount of decolorizing carbon, about the size of a pea, is added to the hot solution. This must be done carefully to avoid a surge of boiling from the hot solution. The solution is stirred and heated for a few minutes and then filtered hot to remove the decolorizing carbon. The resulting filtrate should be colorless and the recrystallization process continues as before.

# 1.6.6 Crystallization the solid:

After the insoluble impurities have been removed, cover the flask containing the hot filtrate with a watch glass and set it aside undisturbed to cool slowly to room temperature. As the solution cools, the solubility of the dissolved compound will decrease and the solid will begin to crystallize from the solution. After the flask has cooled to room temperature, it may be placed in an ice bath to increase the yield of solid. Do not rapidly cool the hot solution by placing the flask in an ice bath before it has cooled to room temperature-this will result in a rapid precipitation of the solid in an impure form because of trapped impurities. Sometimes the dissolved compound fails to crystallize from the solution on cooling. If this happens, crystallization can be induced by various methods. One way to induce crystallization is by scratching the inner wall of the Erlenmeyer flask with a glass stirring rod. This is believed to release very small particles of glass which act as nuclei for crystal growth. Another method of inducing crystallization is to add a small crystal of the desired compound, called a seed crystal, to the solution. Again, this seed crystal acts as a template on which the dissolved solid will begin crystallizing. If neither of these two techniques results in crystallization, the compound was probably dissolved in too much hot

solvent. If you believe that you may have too much solvent for the amount of dissolved compound, reheat the solution to boiling, boil off or distill some of the solvent, and then allow the solution to cool to room temperature again to effect crystallization.

## 1.6.7 Isolating the solid by suction filtration:

Once the compound has completely precipitated from the solution, it is separated from the remaining solution (also called the mother liquor) by filtration. Typically this is done by vacuum or suction filtration using a Büchner funnel. Line the bottom of the Büchner or Hirsch funnel with a piece of filter paper that is large enough to cover the holes in the bottom plate of the funnel without curling up on the sides of the funnel. Place a neoprene adapter on the stem of the funnel and insert it in the top of a filter flask (a thick-walled Erlenmeyer flask with a side-arm) that has been securely clamped to a ringstand. Using a piece of thick-walled vacuum tubing, connect the side-arm of the filter flask to a water aspirator. Turn the water to the aspirator on full force to create a vacuum through the system. If necessary, carefully adjust the piece of filter paper so that it covers all of the holes in the funnel, and then dampen it with a small volume of cold solvent; this will create a better seal between the filter paper and the plate in the funnel, preventing any solid from getting under the filter paper and passing through the funnel. Slowly pour the recrystallization solution into the funnel and allow the suction to pull the mother liquor through. Rinse the Erlenmeyer flask with a small volume of cold recrystallization solvent to remove any remaining solid. Add this solvent to the funnel and then wash the solid in the funnel, called the filter cake or residue, with a few milliliters of fresh, cold recrystallization solvent to remove any remaining mother liquor and dissolved impurities.

Leave the aspirator on for a few minutes and allow air to pass through the crystals to dry them. After pulling air through the crystals for a brief time, remove the vacuum from the system by disconnecting the vacuum tubing from the aspirator before turning the water off. If you turn the aspirator water off first, water can be sucked into the filter flask and may contaminate the product. The filter cake is removed from the funnel by carefully prying it from the filter using a spatula. The cake of crystals will still be slightly wet with solvent and should be allowed to dry thoroughly before measuring the weight or melting point of

# 1.6.8 Objective of the research project:

the solid material. (Frowd, 1998)

The objective of this work is to develop a simple economical method for purification of raw salt which is found in the markets at different parts of the Sudan to be suitable for human consumption as table salt.

## 2. Chemicals and apparatus

## 2.1 Chemical:

Dilute nitric acid 1:1 Analar

Silver nitrate 0.1M

Dilute Hydrochloric acid 1:1 Analar

Oxalic acid (Aldrich)

Conc. Ammonia Analar

Barium Chloride 5% Analar

Buffer solution (buffer 10)

Eric chrome black.T indicator

EDTA solution 0.025 M

Crude salt from the local market

#### 2.2 Apparatus:

Beakers 200 cm<sup>3</sup>, 400 cm<sup>3</sup>, 250 cm<sup>3</sup>

Volumetric flasks 100 cm<sup>3</sup>, 250 cm<sup>3</sup>

Conical flasks 50 cm<sup>3</sup>

Burette 50cm<sup>3</sup>

Pipette 10 cm<sup>3</sup>

Oven

Muffle furnace.

## 2.3 Chemical analysis methods:

Volumetric and gravimetric methods to determine the chloride, magnesium oxide and calcium oxide, While the gravimetric method were used for the determination of SO<sub>3</sub>, CaO and the insoluble impurities.

#### 2.3.1 Purification of the crude salt:

5 gms of the crude salt were weighed in a 250 cm³ beaker. The salt was washed by solution of pure NaCl ((brine)) by adding 20 cm³ of brine solution and stirred vigorously and filtered. Then thefilteratewas left for 10 minutes until the salt settled and filtered through filter paper No 42. The residue was washed with brine solution and dried in the oven.

#### 2.3.2Moisture content:

One gramme of the sample was weighed in a dry and weighed crucible heated in an oven at 105 °C. For one hour. The crucible was left to cool in adessicator and weighed.

#### 2.3.3 Determination of insoluble matter in table salt:

1gm of the sample was weighed and dissolved in 100 cm<sup>3</sup> of water. The solution was filtered through wattman filter paper. The solution was then taken in a weighed crucible and ignited in a Bunsen burner and then in a muffle furnace at 800 °C. For one hour. Cooled in adessicator and weighed.

## 2.3.4 Determination of sodium chloride in the table salt:

0.2 gm of salt was dissolved in 25 cm<sup>3</sup> distilled water in conical flask. I drop of ammonium chromate indicator was added and the solution was titrated against o.1M AgNO3 till the appearance of red colour the volume was recorded and the titration was repeated.

#### 2.3.5Determination of SO<sub>3</sub><sup>2-</sup>:

5 gm of the sample was weighed and dissolved in 25 cm³distilled water and completed to the mark with water.

The solution was taken in 400 cm³beakers, 5ml of HCl 1:1 were added and the solution was heated nearly to boiling, 5 cm³ of 5% BaCl<sub>2</sub> were added and the solution was stirred and left on water bath for one hour, then filtered through N0 42 filer paper. Then the paper and its content, was taken in a previously weighed crucible and ignited on a bunsenburner and transferred to a muffle furnace at 900 °C for 1 hour. The crucible was cooled and weighed to constant weight.

#### 2.3.6Determination of CaO:

1gm of the sample was dissolved in 100 cm<sup>3</sup> of distilled water. The solution was taken in 400 cm<sup>3</sup> beaker, 5 cm<sup>3</sup> of 1:1 hydrochloric acid were added the solution was heated then 10 cm<sup>3</sup> of oxalic acid solution were added, cooled and conc. ammonia was added till the medium changed to alkaline and 5 cm<sup>3</sup> of ammonia were added in excess: The solution was left on a water bath for 1 hour. Filtered

and taken into a weighed crucible and ignited on a Bunsen burner then transferred to a muffle furnace for 1 hour then cooled and weighed.

# 2.3.7Determination of calcium and magnesium volumetrically, using EDTA solution:

1gm of the sample was dissolved in 100 cm<sup>3</sup> of distilled water in a volumetric flask (100 cm<sup>3</sup>), 10 cm<sup>3</sup> was taken in a conical flask, 1 cm<sup>3</sup> buffer 10 was added followed by I drop of Eriochrom black indicator and titrated against the standard EDTA solution till the colour change from red to blue and the volume was recorded.

# 2.3.8Preparation ofbrine solution:

A saturated solution of pure sodium chloride was prepared to be used for removing impurities from the crude salt. The saturated solution was filtered to remove insoluble impurities and was used for removing impurities from the crude salt.

#### 2.3.9 Analysis of samples:

Tow Sample of the crude and purified salt were prepared into 250 cm<sup>3</sup> the analysis were conducted and the results were obtained and the results of the crude, purified salt were compared.

- 2.4 Results and calculations.
- 2.4.1Analysis results of the crude salt.
- 2.4.1.1Moisture Content in crude salt.

Wt of empty crucible + sample= 50.6593 gm

Wt of empty crucible+ sample after heating= 50.6558

Loss in weight

= 0.0043 gm

Wt of sample

= 1 gm.

% of moisture= 0.0043X100 = 0.43 %

1

#### 2.4.1.1 Matter insoluble in waterin crude salt:

Wt. of crucible+ precipitate =29.8578

Wt. of empty crucible =29.8448

Wt. of insol. matter =0.00130

% of insoluble matter= 0.0013X100 = 1.3 %

1

### 2.4.1.2Determination of NaCl in crude salt:

Volume of AgNO<sub>3</sub>= 33cm<sup>3</sup>

No of moles of AgNO3=

$$33 \times 0.1 = 0.0033$$

1000

No of moles of NaCl = 0.0033

Wt of NaCl= 0.0033 X 58 5= 0.1931

% of NaCl =  $\underline{0.1931X100} = 96.5\%$ 

0.2

## 2.4.1.3 % of $SO_3^{2-}$ in crude salt:

Empty crucible= 31.7508 gm

Crucible + sample= 31.7550 gm

Wt of precipitate= 0.0042 gm

Wt of m. wt precipitate in 25 cm<sup>3</sup>= 0.0042 gm

Wt of 
$$SO_3^2$$
 = wt of pptX m. wt of  $SO_3^2$ 

m. wt of BaSO<sub>4</sub>

$$= 0.0042X80X10 = 0.025 \text{ gm}$$

133.33

% of 
$$SO_3 = 0.025X100 = 0.50\%$$

5

#### 2.4.1.4 Determination of calcium oxidein crude salt:

Empty crucible = 29.0897 gm

Empty crucible + CaO= 29.0925 gm

Wt of CaO 
$$= 0.0028$$
 gm

% of CaO in cured salt= 
$$0.0028X100 = 0.0.28\%$$

1

# 2.4.1.5Determination of calcium and magnesium by titration against EDTA solutionin crude salt:

Volume of EDTA = 8 cm<sup>3</sup>

No of moles of EDTA= 
$$8 \times 0.025 = 0.0002$$
 mole

1000

No of moles of Ca and Mg in 10 cm<sup>3</sup>= 0.0002 mole

No of moles of CaO= 
$$0.0028 = 0.00007$$
 mole

40

 $\therefore$  No of moles of Mg = 0.0002-0.00007=0.00013 mole

Wt of MgO = 
$$0.00013X40 = 0.0052$$
 gm

% of MgO = 
$$0.0052 \times 100 = 0.52\%$$

1

# 2.4.2 Analysis Result of refined salt:

#### 2.4.2.1Moisture content in refined salt:

Wt of empty crucible + sample= 50.468 gm

Wt of empty crucible+ sample after heating= 50.466 gm

Loss in weight = 0.002 gm

Wt of sample = 1 gm.

% of moisture= 0.002X100 = 0.20 %

#### 2.4.2.2Matter insoluble in water in refined salt:

Wt. of crucible+ precipitate =29.0758

Wt. of empty crucible =29.0 757

Wt. of insol. matter =0.0001

% of insoluble matter= 0.0001X100 =0.01 %

1

#### 2.4.2.3 Determination of NaCl inrefined salt:

Volume of AgNO<sub>3</sub>= 33.9 cm<sup>3</sup>

No of moles of AgNO3=

$$33.9 \times 0.1 = 0.00339$$

1000

No of moles of NaCl = 0.00339

Wt of NaCl= 0.0033 X 58 5= 0.1983

# 2.4.2.4% of SO<sub>3</sub><sup>2-</sup> inrefined salt:

Empty crucible= 31.8432 gm

Crucible + sample= 31.8457 gm

Wt of precipitate= 0.0025 gm

Wt of m. wt precipitate in 25 cm<sup>3</sup>= 0.0025 gm

Wt of  $SO_3^2$  = wt of pptX m. wt of  $SO_3^2$ 

M.wt of BaSO<sub>4</sub>

$$= 0.0025X80X10 = 0.015 \text{ gm}$$

133.33

% of 
$$SO_3 = 0.015X100 = 0.30\%$$

5

#### 2.4.2.5 Determination of calcium oxid in refined salt:

Empty crucible = 29.0884 gm

Empty crucible + CaO = 29.0896gm

Wt of CaO = 0.0012 gm

% of CaO in cured salt = 
$$\frac{0.0012X100}{1}$$
 = 0.12%

# 2.4.2.6 Determination of calcium and magnesium by titration against EDTA solution in refined salt:

Volume of EDTA = 3.3 cm<sup>3</sup>

No of moles of EDTA = 7X0.025 = 0.0000825 mole

1000

No of moles of Ca and Mg in  $10 \text{ cm}^3 = 0.0000825 \text{ mole}$ 

No of moles of CaO= 0.0012= 0.00003 mole

40

 $\therefore$  No of moles of Mg = 0.000825 - 0.00003 = 0.0000525 mole

Wt of MgO = 0.0000525 X 40 = 0.0021 gm

% of MgO = 
$$0.002 1X100 = 0.21\%$$

1

## 3.1Discussion:

Component	Raw salt	Refined salt	International standards
NaCl	96.5 %	99.1 %	Not less than 98%
CaO	0.28 %	0.12%	Not more than 0.3%
MgO	1.30 %	0.21%	Not more than 0.4 %

SO <sub>3</sub> 2 <sup>-</sup>	0.52 %	0.30%	Not more than 0.5 %
Matter insoluble	1. 30%	0.01%	Not more than 0.1 %
Moisture	0.43 %	0.20	Not more than 0.3%

By comparing the result obtained for the refined salt with those obtained for the crude salt it is obviously clear that the quality of the salt was improved by the treatment which made the refined salt confirmative with the international standards. The percentage of sodium chloride increased to 99.1% which is considered an excellent result. Also the Impurities decreased to the limits which agree with the standard.

The procedure of the purification is simple and easy and not expensive for this reason the purification can be done by the houseWife's if not done by small enterprises. Because the process is simple, not expensive and no loss in salt quantities.

#### 3.2 Conclusion:

The experiments proved the efficiency of brine in removing impurities such as Mg<sup>2</sup>+ Ca<sup>2</sup>+, SO<sub>4</sub><sup>2<sup>-</sup></sup> ion and other contaminants from the salt leading to salt of high purity. The process is simple and can be used by housewife's in villages to purity the crude salt found in those areas to be suitable as food additive.

#### **3.2Recommendation:**

- 1-The authorities should make more inters for the salt now found in the market and standard must be considered.
- 2-small factories should be found to produce table salt which satisfies the standard.
- 3-The consumers awareness, should be raised about the quality of the salt they daily use.
- 4- Iodenized salt should be sold in areas lacking iodine in water and vegetables.

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