INDUSTRIAL CHEMICALS HANDBOOK

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PREFACE

Indian Chemical Industry has developed rapidly in the last two decades. Today most of the industrially important chemicals are being manufactured in India. However, there is still wide scope for the manufacture of a number of new chemicals or for establishing new capacities for the production items in short supply.

Although there are a number of books available on chemicals and chemical technology, most of these books are either too technical or written by foreign authors with their own view point. The present book has been written with a totally practical and Indian view point. The book covers nearly every important chemical and allied item. The processes selected are strictly based on the available sources of raw matreials. Most of the processes dealt with are based on indigeneously available raw materials from sources like mineral deposits in India, agricultural products, sugar factories and down stream products from IPCL and other refineries.

The book includes product profiles of 160 selected chemical and allied products. Each product has been described in sufficient details in simple, lucid and authoritative way. Each product profile includes process flow diagram, chemical reactions involved, raw materials and utilities requirements, process of manufacture, product properties and uses, product market potential, outline of plant economics, equipment and machinery required and major manufacturers of the product.

The plant economics given in the product profiles is in the nature of preliminary guideline for the selection of an item for manufacture and to help in making a rapid initial assessment regarding the project viability. The cost data incorporated in the profiles is of tentative nature as costs of raw materials, equipments etc. are changing from time to time and from place to place. Therefore, the cost estimates given in the book should be taken as guidelines only and they may have to be suitably up-dated and modified before use.

Valuable information have been included in the book in the form of appendices e.g. directory of world-wide contractors of chemical plants, international standards, product-wise directory of chemical manufacturers and dealers in India, ISI standards for chemicals, complete directory of consulting organisations in India and directory of chemical plant and equipment fabricators and suppliers.

It is the sincere hope of the authors that the book will serve as a ready reference and guide to chemical engineers, chemists, manufacturers and all other connected with chemical industry. The authors wish to thank all those who have helped them in the preparation of this book. Suggestions for further improvement will be much appreciated.

AUTHORS

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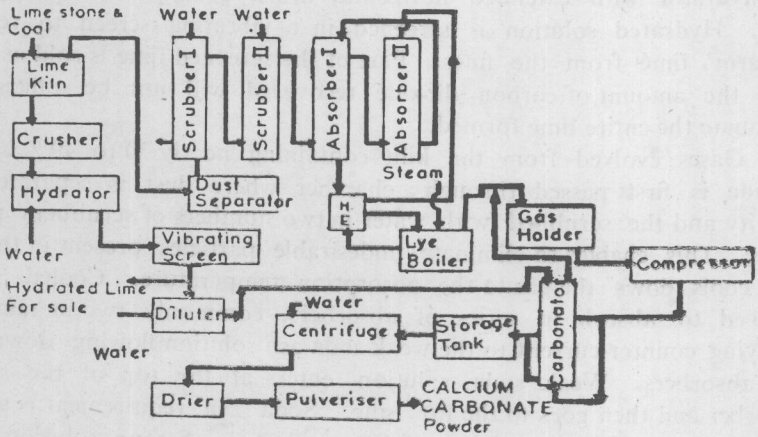
CALCIUM CARBONATE (PRECIPITATED)

$CaCO_3$

By Lime Carbonation Process :



Reaction

$CaCO_3$ (lime stone) $\longrightarrow CaO + CO_2$

$Na_2CO_3 + CO_2 + H_2O \dashrightarrow 2NaHCO_3$

$2NaHCO_3 \longrightarrow Na_2CO_3 + CO_2 + H_2O$

$CaO + CO_2 \dashrightarrow CaCO_3$ (Precipitated)

Raw Material Requirements

Basis : 1 tonne precipitated calcium carbonate

Lime stone (95% $CaCO_3$)	2 tonnes
Soda ash	8 kg.
Coal	600 kg.
Water	10,000 gal.
Electricity	5000 KWH

Process

Precipitated calcium carbonate is manufactured by calcining lime stone and recarbonating the hydrated slaked lime in vertical carbonation towers.

Lumps of lime stone is fed to a hammer mill where it is broken into a size of 10 to 30 cms. and is fed alongwith required amount of coal to a vertical shaft kiln from the top where hot gas from the kiln

preheats the mass. The mass gradually slide down to the calcination zone from the preheating zone. Here the temperature of the material is rised to 1050°±50°C. Calcium carbonate content of the mass decomposes to CaO and CO_2 (decomposition temperature 900°C). The kiln is specially designed to minimise the losses and avoid back reaction which will affect the product quality. The calcined product is discharged down, termed cooling zone of the kiln, where the product is cooled by counter-current air flowing into the kiln, thereby providing heat economy in operation. The air is preheated in this section approximately to a temperature of 300 to 400°C. The quick lime obtained from the kiln is broken to 1" size or finer with the help of a crusher and then slaked in a hydrator with extended horizontal arms, usually in a continuous basis. Hydrated solution is screened in a vibrating screen to remove 'unburnt' lime from the fines. Part of the screened lime is sold as it is since the amount of carbon dioxide recovered will not be sufficient to carbonate the entire lime formed.

Gases evolved from the kiln containing nearly 30 to 40% carbon dioxide is first passed through a chamber where dust is separated by gravity and the scrubbed with water in two numbers of scrubbers put in series. This enables to eliminate undesirable particles present in the gas and cools down the gas to the absorption temperature. Cooled gas is allowed to absorb in series of absorbers, commonly two in numbers, spraying counter-current to the weak soda ash solution flowing downward the absorbers. Weak soda solution enters at the top of the second absorber and then goes to the firt one. Soda ash requirement is approximate 10 to 15 kg. per tonne of CO_2 collected. Strong solution from the bottom of the first absorber is collected in a large boiler/CO_2 stripper. Before reaching the stripper this solution is allowed to pass through a heat exchanger where it is heated up by the recirculated hot weak lye going to the second absorber from the lye boiler. The liquor is boiled by direct heating or by means of closed steam, when absorbed carbon dioxide is released from the bicarbonate solution leaving behind weak sodium carbonate solution. Carbon dioxide thus evolved is collected in gas holders and then sent to the compressor.

The screened lime is diluted in a dilution vessel and pumped to a carbonator constructed of mild steel/R.C.C. and provided with a stirrer. Carbon dioxide is sprayed at the bottom of the carbonator, which will be bubbled through the milk of lime suspension, where it combines with lime to form calcium carbonate. The reaction is exothermic and proper temperature control is maintained to get desired quality of product. The precipitated calcium carbonate suspension from the carbonator is stored in a storage tank and then filtered or centrifuged to get a solid mass of calcium carbonate. The solid thus received is washed in a washing tank to remove undesirable soluble impurities. This will contain 40 to 60% moisture. This is kept in open sun for some time for natural

drying and then dried in air heated tunnel dryer or steam heated rotary drier to get lumps of dried calcium carbonate. Lumps are pulverised in micropulverisers to get fine powder of required mesh size and packed ready for shipment.

From Calcium Chloride and Sodium Carbonate or Ammonium Carbonate :

Reaction

$$CaCl_2 + Na_2CO_3 \longrightarrow CaCO_3 \text{ (ppt)} + NaCl$$
$$CaCl_2 + (NH_4)CO_3 \longrightarrow CaCO_3 \text{ (ppt)} + NH_4Cl$$

Process

In this method calcium chloride, a by-product of ammonia solvay process for manufacture of soda ash, is treated with soda ash (Na_2CO_3) or ammonium carbonate solution to precipitate calcium carbonate. Clear aqueous solution of calcium chloride and sodium carbonate or ammonium carbonate are mixed in a specially designed reactor under controlled process condition of temperature, concentrations and rate of mixing. Precipitated calcium carbonate made by this method is of very high purity. This process is advantageous only when calcium chloride liquor is obtained as a by-product.

As a by-product from Lime-Soda Process for manufacture of Caustic Soda :

Reaction

$$Na_2CO_3 + Ca(OH)_2 \longrightarrow CaCO_3 \text{ (ppt)} + NaOH$$

Process

Here, a 15 to 20% solution of soda ash is treated with milk of lime 90-95°C with agitation approximately for one hour. An alkali solution of 12 to 15% sodium hydroxide concentration is obtained which is separated out. The sludge is concentrated in thickners and the slurry is filtered and washed to get precipitated calcium carbonate.

Out of the above three processes lime carbonation is the most widely adopted method due to the independent nature of this process and due to the availability of lime stone in abundance in our country. Moreover, this process enables effective utilisation of the CO_2 gas evolved during the calcination of lime stone.

Uses

As a filler in paper, plastic and rubber products
Extender pigment in paints and enamels
Manufacture of beauty products
In medicine
Sizing of ropes, textiles, twines and fabrics

Miscellaneous

Properties : Occurs in two crystalline forms—calcite and aragonite.

Calcite : Hexagonal crystals
Mol. wt. 100 M.P. 1359°C at high pressure of the order of 102.5 atm.
Sp.gr. 2.711 (25.$\frac{1}{4}$°C)
Refractive index 1.55

Solubility in water at 25°C 0.0014 gm./100 gm. and at 100°C 0.002 gm./100 gm.

Aragonite : Orthorhombic crystals
Mol. wt. 100 M.P. 825°C (decomposes)
Sp.gr. 2.03
Refractive Index 1.6809

Solubility in water at 25°C 0.00123 gm./100 gm. and at 100°C 0.002 gm./100 gm.

Calcite is formed at lower temperature (below 30°C) and aragonite is formed at temperature above 30°C.

Grades : Light, medium, heavy, C.P., technical, I.P.

Containers and Regulations : Fibre cans, tins, glass bottles, multiwall paper sacks.

Market Potential

Present installed capacity of precipitated calcium carbonate is 27, 870 tonnes per annum, distributed in seven units. Total annual production of these units amount to 22,000 tonnes per annum. Against this, expected demand of precipitated calcium carbonate is 40,000 tonnes per annum whereby a gap of 18,000 tonnes is further to be filled in. The demand of precipitated calcium carbonate is bound to increase further with the progress of plastic and polymer industry based on petrochemical and other process industries.

Plant Economics

Recommended economically viable capacity of above plant is of the order of 5 tonnes per day. Estimated total capital investment required for such a plant is Rs 14 lacs out of which plant and machinery cost amounts to Rs. 10 lacs.

Principal plant and equipment required are

Lime kiln (fire brick lined) ; Dust separator (R.C.C. or M.S.) ; Scrubbers (M.S.) ; Absorbers (M.S. or C.I.) ; Heat exchanger (M.S.) ; Lye boiler (M.S.) ; CO_2 gas holder (M.S.) ; Compressor and transfer pumps ; Lime slaker (M.S.) ; Vibrating screen ; Lime diluter (M.S.) ; Carbonator (M.S. or R.C.C.) ; Srorage tank (M.S.) ; Centrifuge Drier and pulveriser ; Boiler ; One set of pipes, valves and fittings.

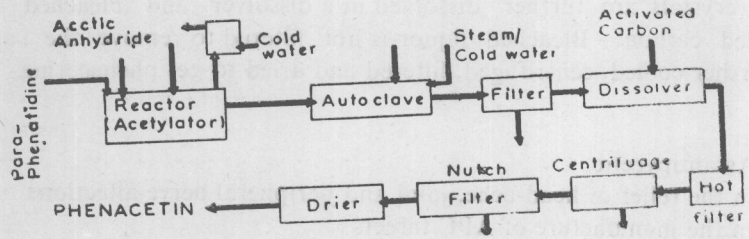
Manufacturers

M/s Burma Lime and Chemical Co. Ltd., Calcutta
M/s Hind Chemicals, Bombay
M/s Radha Chemicals Ltd., Calcutta
M/s Sturdia Chemicals Ltd., Rishikesh, U.P.
M/s Triveni Tissues (P) Ltd., Triveni, West Bengal
M/s SBR Singhania Chemical Industries, Delhi
M/s Winphar Laboratories, Andhra Pradesh
M/s Searsole Chemicals Ltd., Calcutta.

PHENACETIN

$CH_3CONH-C_6H_4-OC_2H_5$

From Para Phenetidine and Acetic Anhydride :



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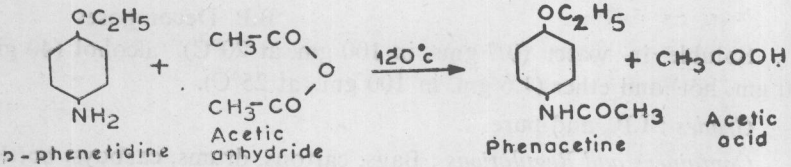
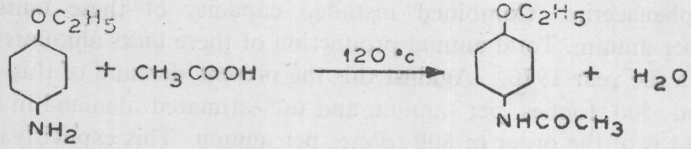




Raw Material Requirements

Basis : 1 tonne of phenacetin

p-phenetidine	0.8 tonnes
Acetic anhydride	0.6 tonnes

Process

Phenacetin is manufactured by the reaction of para-phenetidine with acetic anhydride or acetic acid (glacial, 75% or 50%) in a stainless steel acetylator provided with external jacket and stirring arrangement. When acetic anhydride is used, the entire reaction is divided into two steps. In the first step, phenetidine reacts with anhydride giving phenacetin and acetic acid. Acetic acid thus produced further reacts with a second molecule of phenetidine producing another mole of phenacetine and water.

Calculated amounts of para-phenetidine and acetic anhydride are charged into the reactor with stirring. The temperature of the reactants is kept at 120°C under a reflux. When the acetylation is complete, the reaction mass is transferred to an autoclave where an additional quantity of acetic anhydride is added and stirred with proper temperature control. The reacted mass is then cooled and filtered to get crystals of phenacetin. These crystals are further dissolved in a dissolver and bleached using activated carbon. Bleached liquor is hot filtered to remove the carbon and further cooled, centrifuged, filtered and dried to get phenacetine.

Uses

As antipyretic
In the relief of head-ache, joint and peripheral nerve affections
In the manufacture of APC tablets

Miscellaneous

Properties : Colourless crystalline substance.

Mol. wt. 179.21 M.P. 134°C
B.P. Decomposes

Soluble in water (0.7 gms. in 100 gm. at 20°C), alcohol (40 gm. in 100 gm. hot) and ether (1.6 gm. in 100 gm. at 25°C).

Grades : I.P., and pure.

Containers and Regulations : Bags, cartons, drums, carboys, bottles.

Market Potential

At present there are two units registered under DGTD manufacturing phenacetin. Combined installed capacity of these units is 412 tonnes per annum. Total annual production of these units amounted to 171 tonnes in the year 1976. Against this the present demand of this product is around 500 tonnes per annum and the estimated demand in the year 1983—84 is of the order of 800 tonnes per annum. This explicitly indicates the bitter necessity of putting up new units for the manufacture of phenacetin in our country.

Plant Economics

The recommended plant capacity of an economically viable unit would be 100 kg./day of phenacetin. Total capital investment for such a

unit will be Rs. 7 lacs out of which plant and machinery cost amounts to Rs. 3.5 lacs.

Principal equipment required are

Acetylator (S.S.) ; Reflex condenser (S.S.) ; Autoclave (S.S.); Dissolver (Al) ; Filters ; Centrifuge ; Tray drier ; Boiler ; Transfer pumps (S.S. working parts) ; One set of pipes, valves and instruments

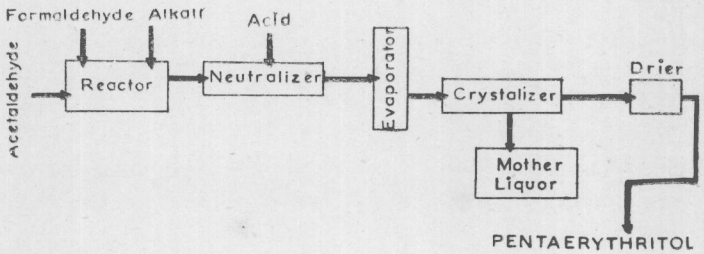
Manufacturers

M/s Aceto Chemicals (P) Ltd., Calcutta-700 001.
M/s Indian Drugs and Pharmaceuticals Ltd., New Delhi.

PENTAERYTHRITOL

$$\begin{array}{l} CH_2OH \\ | \\ HOH_2C\text{-}C\text{-}CH_2\,OH \\ | \\ CH_2\,OH \end{array}$$

From Formaldehyde and Acetaldehyde :



Reaction

$$4HCHO + CH_3CHO + NaOH \xrightarrow[80\text{-}90\%\ \text{yield}]{} C(CH_2OH)_4 + HCOO\,Na$$

OR

$$8HCHO + 2CH_3CHO + Ca(OH)_2 \xrightarrow[80\text{-}90\%\ \text{yield}]{} 2C(CH_2OH)_4 + (HCOO)_2Ca$$

Raw Material Requirements

Basis : 1 tonne pentaerythritol

Formaldehyde (37%)	2.9 tonnes
Acetaldehyde	350 kg.
Alkali (50%)	1 tonne
Acid (as formic)	350 kg.

Process

Pentaerythritol is manufactured by the reaction of formaldehyde and acetaldehyde alongwith an alkali, either sodium hydroxide or calcium hydroxide.

20 to 30% solution of formaldehyde is added alongwith 50% caustic solution or 50 to 80% calcium hydroxide slurry to a reactor with agitation. The temperature of the mixture is maintained at 15 to 20°C. Now 99% liquid acetaldehyde is slowly added under the surface of the formaldehyde-alkali solution. The reactor used is a jacketed one for external cooling of the reaction mass. The heat evolved due to the exothermic reaction is removed by means of cooling water so as to keep the inside temperature of the reactor at 20 to 25°C. The above temperature is maintained for several hours and then raised to about 60°C until the free aldehyde content is less than 0.1%.

The crude reaction mixture from the reactor is transferred to a neutralizer. Here an acid is added to neutralise the excess alkali present and to remove the metallic iron of the condensing agent. If sodium hydroxide is used as the condensing agent, formic acid is added to reduce the pH of the solution to 7.8 to 8. If calcium hydroxide is employed, sulphuric acid or oxalic acid is used to precipitate calcium ion as calcium sulphate or calcium oxalate. In the later case, the solution is filtered after neutralization to remove calcium salt.

The neutralised solution is evaporated up to a specific gravity of 1.27 and further chilled to crystallise pentaerythritol in a crystallizer. The resulting slurry is filtered to get pentaerythritol crystals. The mother liquor is reworked in a recovery system where sodium formate is recovered, when sodium hydroxide is used as the condensing agent.

The crystals obtained after filtration is a mixture of pentaerythritol, polypentaerythritols and formals formed by side reactions. The amounts of these side reaction products can be minimised by proper reaction controls. The crystals yielded from the filtration is ground and marketted as technical grade of pentaerythritol. Pure pentaerythritol suitable for nitration purposes can be prepared from the above by conventional methods like recrystallisation.

Yield is 85—90% by weight based on the acetaldehyde charged.

Uses

Manufacture of alkyd resins
Pentaerythritol tetranitrates (PETN) used as explosives
Upgrading of low cost drying oil
In formulation of varnishes

Miscellaneous

Properties : Colourless crystals or white crystalline powder. Odourless, sweet tasting, neutral and non-hygroscopic.

Mol.wt. 136.15	M.P. 260°C
Sp.gr. 1.399 25°C/4	B.P. 276°C (30 mm) sublimes

Combining weight (number of weight units required to equal one hydroxyl group) is 34.0. All hydroxyl groups are esterifiable. Sublimes