Utilization of Calcareous and other Wastes in the Manufacture of Cement

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About 50 million tonnes (1) of industrial and eral wastes are produced per year from various dustries and mineral processing plants in India. he most important of these wastes from the view oint of cement manufacture are the calcareous wastes obtained from paper, sugar, fertilizer and cetylene industries. Rejects from lime stone quar- es and lime kiln industry, also form calcareous wastes. Data (2) on the availability of some of these lcareous wastes amounting to about 5 million ton- es (mt) are given below in Table 1.

Table 1

Availability of Calcareous wastes

S.No	Wastes	Availability Milliou tonnes per year
1.	Paper sludge	0.5
2.	Phosphochalk (Fertilizer sludge)	1.0
3.	Carbide sludge	1.5
4.	Press mud (sugar Industry)	1.5
5.	Chromium sludge	0.1
6.	Sludge from soda ash	0.2

These sludges are disposed of wet in the form of slurry (moisture content 90 - 95%) or cake (Mo- isture content in the range of 20 - 50%) in settling tanks or nearby places and are considered potential environmental hazards. Calcium Carbonate is the main component of these sludges except in the case of sludge from acetylene industry which produces calcium hydroxide as waste. These wastes are gener- ally fine powders on being dry and may be utiliz- ed as a substitute for limestone in cement manufacture. The constraints coming in the use of these calcar- eous wastes in the manufacture of cement have been noticed and these may be listed as (1) presence of undesirable substances e.g. phosphates, sulphates or alkalies in large amounts (2) high water content as it is in slurry form (3) difficulty in transportation in slurry form (4) involvement of high cost in the beneficiation and drying of slurry. (5) Availability in comparatively in smaller quantity keeping in view the feed requirement of large capacity modern cement plant.

The major raw material for the production of cement is the lime stone having limited percentage of magnesium carbonate (about 5 per cent) in it. Such high purity limestone is generally being kept reserved for steel and other chemical industries. Hence very serious and concentrated efforts are needed to locate and use alternate raw materials other- wise in coming future cement plants may face diffi- culty of raw materials. Therefore, there is an urgent need in the utilization of these sludges by developing appropriate technology for the manufacture of cement.

Small amounts (5 - 10 percent) of these sludges as a supplement or sweetner for upgrading the quality of limestone in few cement plants are being used in India. Vachani(3) showed the utility of lime sludge from a PVC produced 2 tonnes of dry lime sludge - $Ca(OH)_2$ is produced.) A plant in Kota(4) based on the lime sludge from acetylene industry, high grade chips and low grade limestons deposits available in the vicinity is being set up having a capacity of 200,000 T/year. Paper sludge is also used on a very limited scale in cement industry. The problems faced in the use of paper factory sludge has been descri- bed by Jain & Malhotra (5) while working on this as (a) Moisture & quality of sludge not uniform (b) great variation in the alkali content in the sludge (c) high percentage of NaOH & Na_2S in sludge which in turn have adverse effect on lining plates & re- fractories. (d) Refractory bricks accordingly to be changed by chromium magnesite bricks & other high alumina bricks. They have opined that judicious without compromising on quality in any way. These bricks are also ideal for application, where high strength is required, like in foundation or while building bridges.

Another important field of klinker-application would be for surfaces exposed to acids or other chemical hazards as well as for canal lining and other constructions exposed to flowing water.

See fig. 5 for a typical klinker-faced building

This system is also utilized for the manufacture of glazed and unglazed tiles also using the Spengler press and suitable brick clay. This is possible because the filling height of the mould boxes can be adjusted between 25-250 mm.

In a tile-plant, one line of production could be unglazed vitrified tiles. Ideal sizes are 200×200mm and 200×100 mm. Thickness can be varied from 9-12 mm for wall and floor-tiles and around 20 mm for heavy-duty industrial floor tiles and also for other usages like pavement tiles, canal lining etc.

In a sophisticated plant, the wall and floor-tiles could be glazed. Further, the glazing can be done in 2 systems.

One system is the dry-press-glazing or rustical glazing. Here the glazing is done at the press itself. An attachment at the press strews dry glaze on the top of the press-body in the mould-box. While the tile is being pressed, the glaze is pressed too into the surface. The glazing effect is uniformly random rustical.

The other system is the classical wet-glazing system by spraying and water-fall methods. Unglazed tiles from the press are dried in a rapid drier and then glazed on a glazing line.

In a sophisticated tile plant, the granulation, however, is carried out in a spray-drier. The firing should be in a rollerkiln or a single-layer kiln, fuelled by clean producer gas or LPG.

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use of sludge requires uniform quality in the first instance. Mall & Prakash (6) on the other hand, working on the use of sludge from Kraft paper Mills & opined that effective wasing of sludge can reduce alkali content in it. There will be no clogging & ring formation in the kiln during the clinkering. They concluded that by using only 5 per cent lime sludge in the raw mix there is no difficulty in the manufacture of cement and process becomes free of any problem. To reduce the effect of alkalis fluorogypsum has also been tried.(7) Chemical composition of the sludges vary a great deal from industry to industry and may be summerised(2) from various industries in Table 2 as follows :-

manufacture of cement are phosphogypsum, flurogypsum and red mud. Phosphogypsum and flurogypsum are reported (8) (9) to act as mineralizers in the manufacture of cement beside their use as setting time controller for the finished product. There are seven plants in the country producing phosphogypsum and two producing flurogypsum (anhydrite calcium sulphate from hydrofluoric acid manufacture). Four more plants are being set up to produce phosphoric acid which in turn will produce phosphogypsum as waste. The availability of usable phosphogypsum is expected to be around 5 million tonnes per year which has the potentential of being used in building material industry if appropriate technologies for

Table 2

Chemical Composition of different sludges

	Percentage					
Constituents	paper	Sugar	Fertilizer	Chromium	Soda Ash	A cetylene
LoI	35-40	40-50	34-38	20-35	34-38	25-30
SiO_2	4-6	2.5-4.5	3-5	4-6	1-2	4-6
Al_2O_3	2-5	2-5	0.3-0.5	3-5	1.5 3.0	1-3
Fe_2O_3	1-1.5	2-2.5	0.3-0.5	-	1-2	0.1-0.25
CaO	45-50	42-50	45-50	35-40	44 48	60-70
MgO	1.5-2.0	4-10	-	3-6	1-2	0.2-0.5
SO_3	-	1-2	8-10	-	-	0.2-0.3
P_2O_5	-	-	1.5-20	-	-	Tr
F^-	-	-	1-2	-	-	-
Cl^-	-	-	-	-	6-10	0.2
Na_2O/K_2O	0.5-1.5	1-2	-	1-1.8	-	0.02-0.2
Cr_2O_3	-	-	-	18-20	-	

It may be observed that all the sludges contain $CaCO_3$ as a main constituant except waste from carbide industry which contain calcium hydroxide. Beside this there are other constituents as impurities which become part of the Sludge during the process which is employed in particular industry. Paper, Sugar and Chromium Sludges contain alkalis (generally more than 1 per cent) which are not desirable in raw materials to be utilized for cement manufacture.

The other wastes which may find use in the

its utilization are available.

Red mud is a residual waste obtained from aluminium industry. For each tonne of aluminium prodused nearly 0.9 tonne red mud is generated. It is in fine form. The availability of red mud is about 1.5 million tonnes per year.

The typical chemical analysis of phosphogypsum, flurogypsum & red mud are as follows (10) Table 3):

Table 3

TYPICAL CHEMICAL ANALYSIS OF SOME WASTE

Constituents	Percentage Phosphogypsum	Flurogypsum	Red Mud
LoI	30		5-7
SiO_2	1.11 - 3.94		20-30
$Al_2O_3+Fe_2O_3$	0.91 - 1.91	Same as for Phosphogypsum	30-40) Fe_2O_3 15-20) Al_2O_3
CaO	31.39 - 33.08		0-1
MgO	0 .8		0.1
SO_3	43.07 - 45 03		-
F-	0.5 - 1.0	1 - 1.5	-
P_2O_5	0.49 - 0.88	-	-
Alkalis	0- 0.23		1-2

The chemical analysis of all the above wastes reveal that red mud has potential to be used in place of clay or as a part substitute to clay in cement raw mix. Phosphogypsum have potential to be used in place of mineral gypsum as a set controller of cement and for its possible use as mineralizer cement raw mix.

The extent of suitability of these sludges and other wastes would ultimately depend upon their composition, percentage of deleterious constituents and possible stabilization of these harmful constituents. in these wastes.

ALKALIS :

The Alkalis present in the cement raw mix effect the coures of reaction in the kiln in the form of chemical attack on the refractory lining[11] and properties of finished product. Formation of lumps in cement during storage has been attributed to the formation of alkali phase, syngenite[12] ($K_2SO_4\,CaSO_4\,H_2O$). It has also been observed that alkali silicates & aluminates formed during clinkerization in alkali rich materials are more sensitive to reactions with H_2O and CO_2 present in air and presence of these phases could be one of the causes of formation of lumps during cement storage[13].

The alkalis may also react [14] with dicalcium silicate and tricalcium aluminate. This have the effect of enlarging the CaO primary phase region by the reactions like—

$$12\ C_2S + K_2O = KC_{23}S_{12} + CaO$$

OR

$$3C_3A + Na_2O = NC_8A_3 + CaO$$

To avoid the adverse effect of alkalis [15], SO_3 is added. Alkalis react preferentially with SO_3 and forms alkalisulphates. This reaction reduces the availability of K_2O and Na_2O in the reaction to form $KC_{23}S_{12}$ and NC_8A_3 so that the potential phases present in the clinker remain nearly the same as to be obtained with low alkali content. Both NC_8A_3 and $KC_{23}S_{12}$ react with calcium sulphate.

$$Na_2O\ 8CaO\ 3Al_2O_3 + CaSO_4 = Na_2SO_4 + 3\ C_3A$$

$$K_2O\ 23CaO\ 12SiO_2 + CaSO_4 = K_2SO_4 + 12\ C_2S$$

It is, thus, only the alkalis in excess of this requirement which will form NC_8A_3 and $KC_{23}S_{12}$. Sodium and potassium sulphates form a solid solution. New Kirk [16] found that solid solution approximating a composition to 3 K_2SO_4:Na_2SO_4 had formed. The content of the SO_3 (0.1-0. 5per cent) is not normally sufficient to satisfy all the alkalis in the mix composition. Alkalis are usually present in excess of SO_3 & the surplus alkali forms N C_8A_3 & K $C_{23}S_{12}$ while some of it taken up by C_2S and C_3S. Presence of alkalis plays an important part in the rate at which calcium silicates react with water.

In majority of the cements the content of K_2O exceeds that of Na_2O and the sulphate formed then tends to have a composition ([17]) around $K_{0.75}$ $Na_{0.25}$ SO_4. Presence of alkalis only affects the C_3S content of a cement by the order of 1 or 2 per cent but C_2S content is substantially affected ([18]). Excess of SO_3 may give a saturated solution of Ca SO_4 in C_3S which contain about 2.9% SO_3 ([16]). But sulphatic C_3S has lower hydraulicity and the strength decreases with increasing SO_3 content ([18]). To decrease the effect of alkalies on the quality of clinker Moskvin et al ([7]) used 1-5% flurogypsum containing CaF_2 3.13% and 85% CaS04.2H 0. Firing at 1400° for 1 hour showed that with 5 per cent flurogypsum practically complete bonding of the alkalies as sulphate takes place and clinker activity increased by 16 times. So it may be noticed that chemical remedies are possible to reduce the effect of alkalis but with restrictions.

PHOSPHASES— The content of P_2O_5 in most of the cement is of the order of 0.2 per cent. At low levels ($\approx$ 1%) P₂O5 acts as a mineralizer. But if phosphate occurs in large amounts in cement raw mixes or raw materials being used for cement manufacture e.g. sludges it is retained into the clinker. The effect of this is that the rate of hardening of cement so produced is slow. This is because P_2O_5 decomposes C_3S in favour of C_2S solid solution containing P_2O_5 and free CaO. At lower concentration solid solution will contain C_3S & C_3S'' as a solid phase on clinkering while at higher P_2O_5 contents the stable phases become, lime & C_2S-C_3P solid solution. Since a portland cement must contain an optimum portion of C_3S, the maximum proportion of P_2O_5 in a cement mix must not exceed 2.0-2.5 per cent ([19]). Nurse ([20]) has opined that P_2O_5 decomposes, C_3S forming a series of solution between C_2S and 3 CaO P_2O_5 he found that for each 1 per cent P_2O_5 added to a cement mix, the C_3S content is lowered by 9.7 per cent and the C_2S (now a solid solution) raised by 10.9 per cent. He also showed that satisfactory cement can be manufactured by cement clinker containiug upto 2.5 per cent P_2O_5 by using correct burning (short burning time and rapid cooling) and proportioning but the rate of hardening was slow. Steinour ([21]) suggested use of fluoride to overcome the harmful effect of phosphates. Russian workers ([22]) suggested the use of Cr_2O_3 to manufacture cement from raw materials containing phosphates.

SULPHATES :

The behaviour of sulphate ions is different than phosphate ions. It increases the formation of BC_2S solid solution rich in aluminium and sulphate and does not decompose C_3S but C_3S content is decreased in preference to C_2S in the system CaO - SiO_2 - Al_2O_3 - SO_3 and in cement clinker without magnesia and alkalis. Magnesium ions counter-act the adverse effect of aluminium and sulphate ions on C_3S formation. On the other hand ferric ion and sulphate ions together do not hinder C_3S formation([23]) even in the absence of magnesium ions. The phases generally formed during clinkerization are C_3A_3 Ca-SO_4 ; 2 (C_2S), CaSO4; 3(C_2S) - Ca SO_4 and Ca SO_4 1.75 SiO_2. Presence of sulphate ions when SO_3 4 per cent it causes sulphate expansion and is detrimental to strength development. Overall effect of sulphate (24-26) is that it acts as effective mineralizer, modifies alkali recycle by forming less volatile compounds (N, K/SO4 compounds when S (K+N), increases belite content, decreases hydraulic and mechanaical strength and causes sluphate expansion when present in high percentages (generally more than 3 per cent SO_3 but depending upon the mineralogical composition of cement) Modified cements containing C_4A_3S and C_3S formation have been reported([27]) but their long term performance behaviour and durability needs to be reestablished.

CHLORIDES : If chloride is present in an amount more than 0.02 per cent in limestones, it is not regarded suitable for manufacture of cement because corrosion of reinforcement in the concrete structure may take place. However the limiting range in a cement raw mix is 0.6 per cent. Higher Cl^- forms more volatile (K, N) Cl and causes operational problems due to vaporization in the burning zone. The chances of ring formation are increased. A by-pass of the exist gases is required if Cl is present in more than .015% [28] [29].

A special cement having alinite phase has been reported [30] [31] to be manufactured by the use of high amounts of chloride but its use and durability are still to be established beside this it may have limited use only.

MAGNESIUM OXIDE: The presence of MgO within limits of upto 5 per cent in cement raw mix is desirable from the view point of first appearance of liquid. Addition of MgO to alkali containing materials also accelerates lime assimilation [32]. However high content of MgO leads to delayed expansion of cement (due to formation of periclase crystals during clinkerization) causing damage to concrete structures. When gypsum is used as a mineralizer in presence of MgO, stability of C_3S phase on cooling results or when Al^{3+} & SO_4^{2-} can impoverish the clinker of C_3S [34]. Presence of SO_3 in an amount of 0.67 per cent when MgO is equal to 2 per cent neutralization effect of volume instability has been also reported [28] [34].

CONCLUSION

The use of calcareous and other wastes as components of cement raw mix or as a substitute of conventional raw materials has recently assured great importance. The reasons behind this awareness are rising costs of these wastes. Due to the impurities present in the wastes and their effect during clinkerization and on cement so produced their use require certain precautions. It is, thus may be concluded that (1) the effect of impurities present in the sludges as discussed above may require beneficiation (2) these wastes may be used to manufacture special cements (3) certain impurities present in the sludges possess mineralizing action and this aspect may be utilized by adopting suitable raw mix design and (4) these wastes may be used for the manufacuure of ordinary portland cement by suitably stabilizing the undesirable constituents in sludges and other wastes.

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Major reliefs for cement proposed

A sharp hike in the retention price, remission of excise duty and higher non-levy sales are among the major reliefs to the cement industry being considered by the Union cabinet.

The reliefs are intended to meet the industry's rising costs and to make its operations more economically viable.

The cabinet's proposal is to extend the concessions to units being set up in the seventh plan as well as to those established between 1982 (when the partial decontrol scheme came into force) and 1985. The concessions will also be applicable to expansion schemes.

The relief contemplated for units which have started production during the seventh plan is around Rs 90 to Rs 100 a tonne of which the retention price (levy price) may work out to about Rs 40 a tonne and the balance in the form of excise duty rebate etc.

Nearly 60 per cent of the relief proposed for seventh plan units may go to those established between 1982 and 1985. The latter is expected to receive about Rs 50 a tonne by way of relief—Rs 40 a tonne in the form of hike in the levy price and the rest in the form of excise rebate etc.

Of the proposed retention price increase of around Rs 40 a tonne, Rs 24 a tonne represents the cost escalation till September, 1986 (since the last revision in July, 1984).

The balance of about Rs 16 a tonne is by way of an additional non-levy quota of five per cent (Rs 11 a tonne) and abolition of the obligation of cement units to contribute Rs 9 a tonne on their non-levy sales to the cement regulation account (about Rs 5 crore).

A novel feature of the praposal is that it does not envisage any fresh burden on the government, the main consumer of levy cement. The price increase is proposed to be met out os CRA, which has now about Rs 150 crore.

The funds are in the from of freight contributed by manufacturers for movement from one area to another. The freight is calculated at Rs 180 a tonne against weighted average of Rs 160 a tonne.

It is felt that this cushion of Rs 20 a tonne can be set apart to meet the proposed increase in the levy price. The total levy obligation on the industry for 1986-87 has been fixed at 18 million tonnes. On this basis, around Rs 50 crore may have to be withdrawn from CRA.

The removal of the obligation of cement units to contribute Rs 9 a tonne on their non-levy sales to CRA, bringing the total amount to Rs 70 crore.

The proposal to give wideranging reliefs to the cement industry had been mooted by the new minister for industry. Mr J Vengala Rao, who is keen on the cement industry meeting its production target by timely revision of price etc. to meet cost escalations.

Industry circles, however, feel that the proposed reliefs may not help the industry obtain the 12 per cent post-tax return promised to the industry under the partial decontrol scheme. The return may at best be about seven or eight per cent.

Again the price increase contemplated does not take into account the latest increase in railway freight. According to these circles, the industry ministry should initiate immediate steps to overcome the cement industry's infrastructural constraints to enable it to hit this year's (1986-87) production target of 36 5 million tonnes.

As per the latest estimate, total production in the first seven months had been only around 19.5 million tonnes (including that of mini cement units). This means that in the remaining period, production has to be about 3.5 milllon tonnes a month so that the target is achieved.

It is understood the industry minister proposes to call a meeting of cement manufacturers shortly to review the production performance and to take urgent steps to meet their infrastructural problems.

Multi-modular system to move cement

The government is evolving a new system for movement of cement by rail, road and waterways to ensure adequate availability all over the country, the Union minister for industry, Mr J Vengala Rao said.

The government would also do everything possible to maintain the health and progress of the cement industry as evidenced by the measures announced recently, he statad.

Mr Rao was inaugurating a fiveday international seminar here on "pragmatic strategies for productivity and modernisation" as related to cement and building materials industries. The seminar was organised by the National Council for Cement and Building Materials (NCB).

Stressing the need to adopt a modern bulk distribution system for cement, he felt the existing arrangement for bulk loading was antiquated and crude and could not withstand the requirements of the unprecedented growth visualised for the industry.

This deficiency was most conspicuous in the light of the progress made by other countries which had expanded their markets and rationalised the distribution system by introducing extensive technological innovations.

He was, however, happy that under NCB's aegis considerable progress had been made in developing systems appropriate to Indian conditions for bulk transport of cement. A pilot study for bulk movement by rail in the Bombay-Wadi corridor had been entrusted by the development commissioner for the cement industry to Rites along with NCB. Depending on the success the study could be extended to other viable corridors.

Similarly, in the area of packing of cement, substantial improvement and development had taken place due to NCB's initiatives and the jute industry, plastic industry, bag manufacturers and the cement industry