

Utilization of Nano Technology in Reducing Moisture Content of Phosphate Cake at RSMML Rock Phosphate Beneficiation Plant

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Abstract

After the induction of High Pressure Grinding Rolls at rock Phosphate Beneficiation Plant, The belt Drum Filters (BDF's) performance rapidly deteriorated. This was mainly attributed to fine grinding i.e., about 85% < 25 microns. The phosphate cake produced by BDF was sloppy and posed problems in handling and transportation. The sloppier the product the more inefficient in loading and transportation¹. Thus the most daunting task was to reduce moisture content of phosphate cake from about 22% to 13-15%. This paper discusses the most critical role played by nanotechnology in tackling this problem.

BACKGROUND

RSMML has been operating Belt Drum Filter (BDF) since the inception of the rock Phosphate Beneficiation Plant. BDF performance had been satisfactory. BDF's were usually operated at 40-50 Mt/hr. The cake discharge was satisfactory with the moisture content of about 15 %. After commissioning of High Pressure Grinding Rolls, the Plant capacity has been doubled. BDF's are now operating at 70-80 Mt/hr but the moisture content of the cake is on the higher side i.e. 20-22%. Wet cake is posing problems in material handling and transportation. Vigorous efforts were made to reduce the moisture content by plugging leakages at various locations like hyflow valves, drainage tubes and stainless steel nipples etc. But unfortunately, despite of all possible efforts, the situation remained more or less the same. Thus the problem at this stage was to find an optimal condition for efficient filtration where the moisture content could be in the range of 13 to 15 %. It was realized that the phosphate feed consisted of very fine particles which posed problems during dewatering stage in filtration. The dewatering of phosphate cake was not very efficient.

It was recognized that some steps would have to be taken to increase phosphate cake permeability without increasing its porosity. Thus the role of nanotechnology was envisaged². Manipulation at macro level was done by bringing in the macro molecules. The role of Macromolecules which can hold together large number of suspended particles into a single entity, which settle down rapidly due to gravity i.e. flocculent was assessed. Utilization of these macromolecules i.e. polyelectrolyte in thickening and filtration gave some very interesting results.

INTRODUCTION

Nanotechnology now a days find wide application in various fields like pharmaceutical, paper, textile, mining and mineral beneficiation, and ceramic industries etc. Nanotechnology is the art of manipulating materials on a very small scale. Macromolecules of several polymers containing ionogenic groups are capable of dissociating into ions in solution³. Such macromolecular electrolytes or polyelectrolyte do wonder at thickening and filtration processes. In order to improve the thickening and filtration efficiency at Jhamarkotra Rock Phosphate Beneficiation Plant, polyelectrolytes were induced at phosphate thickener and at the pulp distributor of BDF. Polyelectrolytes molecules bring together the large number of suspended particles by forming bridges between them. In the bridging mechanism, the polymeric chains form bridges among suspended particles and create flocs which sediment by gravity.

A BRIEF REVIEW OF POLYELECTROLYTES

Stable suspension may be flocculated, by addition of certain organic polymers of high molecular weight. These are known as “poly-electrolytes”. Mixture of lime and starch, glue and gelatin are also used as flocculants. Water soluble polymers can be classified according to their sources, their constitutions and /or the ionic properties they exhibit in aqueous media⁴. See Table-1.

Table 1: Classification of Flocculent

Source	Type		
	Non- ionic	Anionic	Cationic
Natural	Guar gum Starch	Alginates	---
Modified natural	Dextrin	Causticized starch Carboxy methyl cellulose lignosulphonates	----
Synthetic	Polyacrylamide Poly ethylene oxide Poly vinyl alcohol Alkyl hydroxamate	Hydrolyzed polyacrylamide Sodium polyvinyl sulphonate Sodium polystyrene sulphonate	Polyethylene imine Polyacrylamide Poly vinyl pyridinium bromide

Polyelectrolytes are widely used as flocculents⁵. The most effective flocculating agents are polyelectrolyte having sufficiently high molecular weights. Flocculation may occur because long chains of polyelectrolyte molecules are absorbed by one end on one suspended particles, and by other, on another particles, forming a sufficiently strong bridge between particles.

Various types of flocculents are now being produced on the industrial scale. One of the most widely used flocculent is partially hydrolyzed polyacryl amide⁶⁻⁷, which contain the -NH₂ and -COOH groups. When the degree of hydrolysis is higher, the polyacryl amide molecule acquires a negative charge which is so high that the molecule is no longer absorbed on the negatively charged particles of suspensions.

The bridging mechanism indicates that with increase in molecular weight of a polymer the efficiency of flocculation increases, since a longer chain bridge more effectively. Numerous investigators have reported evidence supporting this hypothesis. The lower molecular weight polymers can also bridge particles provided that the surface charge is low enough for the close approach of the particles, or if the absorbed polymer reduces the potential by charge neutralization, thus reducing interparticle repulsion. Various important parameters of flocculating system are listed in **Table-2**.

Table 2: Variables of Flocculating System

S.N	Properties	Likely observed and effects
1.	Molecular weight increase	Poor solubility, high viscosity solution, chain more shear sensitive, higher unit cost, high optimal dose, higher unit cost, bridging favored, larger flocs, higher sediment volume, higher water retention, faster settling.
2.	Dose increase	Better flocculation up to optimal dosing.
3.	Shear increase	Break down of long chain, irreversible floc degradations smaller equilibrium size flocs.
4.	Particle surface area increase	Increased flocculant consumption and ultra fines susceptible to overdosing.
5.	Bridge concentration increase	Does not always affects optimal dose, smaller and longer flocs.

APPLICATION OF POLYELECTROLYTES IN MINERAL BENEFICIATION PLANTS

Flocculent is widely used in enhancement of sedimentation and filtration. Sodium polyacrylates, polyacrylamides and hydroxamates function groups are used in Alumina plant for red bud settling⁸. Polyelectrolytes True flocks and Coliflock⁹ are used in coal washeries for sedimentation. Similarly Polyelectrolytes Super floc 100 and Magna floc 1011(anionic) were found to be very effective in settling of solids, in preparation of pulp, prior to leaching of Uranium ore¹⁰. Polyelectrolytes are used as flocculants for coagulating circulating water in coal industries and extracting gold from wash and wastewater in the gold extracting industries. It is used in paper industry for retaining the filler in paper and reducing fibre losses. It is also employed for purifying wastewater. Its various application are shown in Table3.

Table 3: Application of Polyelectrolytes

S.N	Name of industry	Objective
1.	Textile effluent	Flocculation of coagulated coloring matter
2.	Pulp & Paper dewatering	Separation of pulping waste water & sludge
3.	Coal Dressing	Sedimentation
4.	Dyes & chemicals	Effluent treatment
5.	Sludge waste	Dewatering.
6.	Ceramic/pottery	Sedimentation & dewatering.

Although flocculent are used for sedimentation and filtration. However, the specific requirements of a flocculent used to promote sedimentation are not necessarily the same as for the one used for filtration. Dense flocs promotes settling and are suitable for clarification, thickening and sedimentation where as large flocs are desirable for filtration as de-watering is quite efficient Therefore optimization of solid/liquid separation process require careful control of floc size and structure.

The factors influencing the degree of flocculation are as under:

- a) Efficiency or strength of adsorption of the polymer on the surface.
- b) The degree of agitation during flocculation and subsequent agitation, which can result in breakdown of flocs.

The maximum effect of flocculent is achieved at an optimum dosage rate. Excessive polymer dosing can cause dispersion of particles due to floc breakdown. Particle collisions and hydrodynamic interaction are also of great importance, growth and development of flocs effecting physical factors. Even pumping of the flocculated slurry may destroy the floc due to rupture of long chain molecule. Much can be done to improve the dewatering capability of concentrate by conditioning with flocculent. This has been subjected to intense investigation¹⁰ since from early 1970's.

APPROPRIATE PULP DENSITY AND PARTICLE SIZE DISRIBUTION OF THE FEED FROM PHOSPHATE THICKENER

Moisture content of phosphate cake depends upon the pulp density of the slurry and up to some extent the size distribution of the particles. The pulp density should be in the range of 1.4 to 1.6 gm/cc. A representative sample of feed to filter from phosphate thickener was analyzed on standard sieves for particle size distribution. Table-4, shows the particle size distribution of the feed. This clearly indicates that it is composed of predominately finer size i.e. 75% below 25 microns. Presence of large amounts of silica particles in finer fraction makes things even worse. The filtration of such feed material becomes arduous task due to almost absence of coarse particles. To overcome this, it was initially visualized that formation of large size flocs may enhance filtration rates along with low moisture content of discharge cake. Thus utilization of flocculent was envisaged.

Table 4: Sieve Analysis and Chemical Composition of Phosphate Feed to BDF in Absence of P.E

Mesh No.	Size In microns	Weight retained (gm)	(%)	Chemical composition		
				P ₂ O ₅	SiO ₂	MgO
+200	+74	8.6	2.87	12.6	24.1	8.70
-200+300	-74+53	18.2	6.07	12.40	20.95	9.50
-300+400	-53+37	21.7	7.23	16.70	15.16	8.80
-400+500	-37+25	22.9	7.63	26.10	11.68	5.0
-500	-25	228.6	76.2	38.0	3.37	0.80
Head sample				32.60	6.82	2.70

EXPERIMENT DESIGN

- a) For selection of proper electrolyte, Jar test was conducted by considering 3nos.of anionic Polyelectrolyte viz., Max floc A-107, Max floc-A-108 and SS-120.The Jar test observation is shown in Table-5.Cationic Polyacrylamide C-21 & C-33 were also tested.

Table 5: Jar Test Data-Comparison of Various P.E in Settling Phosphate Slurry (Feed from Phosphate Thickener)

S.N	Name of P.E	Dose (ppm)	Time (min)	Suspended water quality	Sludge volume
1.	P.E max floc A-107	5	15	Clear	425
2.	P.E max floc A-107	9	15	Clear	420
3.	P.E max floc A-108	6	15	Clear	315

- b) Turbidity at phosphate thickener (P.T) over flow (O/F) water was also measured with and without P.E. P.T O/F water sample was collected prior to addition of P.E. Max floc A-107 at the sump feed of feed P.T D.C drive pump. After 6 hours another sample was drawn. Analysis of these samples is shown in Table-6.

Table 6: Analysis of Phosphate Thickener Overflow Water in Presence /Absence of P.E Max Floc A-107

S.N	Sample description	pH	Total suspended solids	Turbidity (N.w)
1.	Phosphate thickener over flow water in absence of P.E	5.8	74	89.50
2.	Phosphate thickener over flow water in presence of P.E	5.8	72	47.00

- c) To assess the impact of P.E on moisture content of the discharge cake, performance of only BDF 3002 was taken into consideration. This has enabled us to predict various factors, which are playing vital role in flocculation. Most of the variable parameters like vacuum pressure at BDF, rpm of Drum, pump rpm etc were assumed to be constant. The vacuum pressure was usually in the range of 400-450 mm of Hg. In order to analyze performance of P.E, average moisture content in shift versus number of observation i.e. counts (3 per day) were plotted. This curves were plotted for the following cases:

- Reference plots i.e., no P.E addition, see fig.1.
- Addition of P.E (SS-120) at BDF distributor box only, see fig-2.
- Addition of P.E at feed to P.T pump (Max floc A-107) as well as BDF distributor box (SS-120) see fig-3.

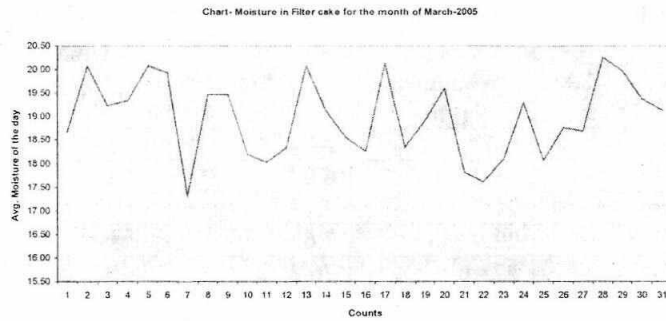


Fig. 1: Average moisture content of phosphate cake in absence of P.E. (reference plot)

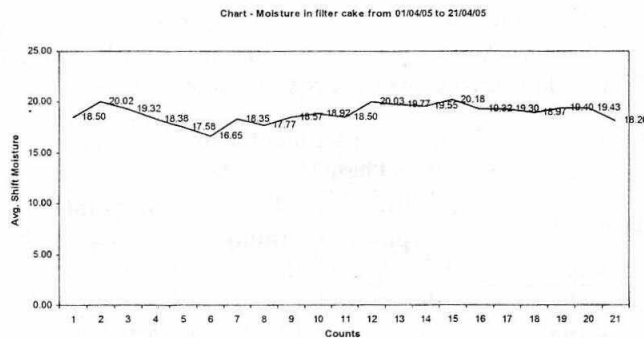


Fig. 2: Average moisture content of phosphate cake in presence of P.E. SS-120 at BDF distributor

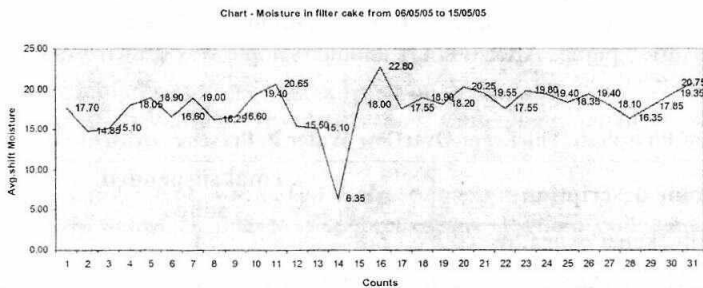


Fig. 3: Average moisture content of phosphate cake in presence of P.E. SS-120 and MaxflocA-107 at BDF distributor & P/T respectively

RESULT AND DISCUSSION

The results of this work could be broadly summarized as:

I. Impact of P.E Doses at Thickener/Filter

- BDF performance in absence of P.E-Figure-1 shows the variation in moisture content of discharged cake with time. The average moisture content without P.E addition is on the higher side i.e. 18.97.
- BDF performance in presence of P.E (SS-120 & Max floc-A-107).

Figure-2 shows that there is some significant impact on the average moisture content of the shifts .A average moisture content decreases up to 16.65 and then again increases. It never remains low for the very long duration of time. This may duet either some mechanical snag at BDF or also inefficient de-watering of the filter cake.

- c. BDF performance in presence of P.E at multiple dosing points i.e. at phosphate thickener & BDF distributor box. Flocculent at multiple points was added i.e., P.E Max flocs A-107 and SS-120 at phosphate thickener feed to P.T sump and filter distributor box respectively. Dry cake at similar size distribution (see table.7) was observed. Figure-3. shows a drastic dip in the moisture content. This may be due to faster sedimentation at phosphate thickener as well as highly efficient filtration. Addition of higher molecular weight strongly anionic P.E at phosphate thickener promotes rapid thickening and sedimentation.. The higher the molecular weight, better is the flocculation and faster is the sedimentation. The application of lower molecular weight P.E SS-120 appears to have assisted in large size floc formation. These have higher resistance to shear. The resulting filter cake is a uniform porous structure, which allows rapid dewatering, yet prevent migration of the finer particles through the cake to the filter medium. This clearly established that dewatering of cake is very efficient when P.E is given at multiple locations. Moisture content increases after producing about 4000 Mt of dry phosphate concentrate between 6.05.05 to 13.05.05., having moisture content of about 14 to 17%. Then after the moisture content again increases due to leakage's at drain tubes, hyflow valve, and steel nipples.

Table 7: Sieve Analysis and Chemical Composition of Phosphate Feed to BDF in Presence of P.E

Mesh No.	Size in microns	Weight retained (gm)	(%)	Chemical composition		
				P ₂ O ₅	SiO ₂	MgO
+200	+74	5.2	1.73	-	-	-
-200+300	-74+53	10.6	3.53	16.2	29.07	5.10
-300+400	-53+37	14.4	4.8	23.20	25.52	3.0
-400+500	-37+25	20.4	6.80	30.40	17.69	1.40
-500	-25	249.4	83.13	37.9	4.48	0.60
Head sample				35.8	7.92	1.20

- d. Table-6., shows that P.E max floc A-107 addition improves clarity of over flow water at phosphate thickener. This is due to flocculation of fine sized phosphate particles. Dense flocs are formed and settle down rapidly. Thus the turbidity of phosphate thickener overflow water is lowered i.e., 89.50 to 47N.w.
- e. Table -5. Jar Test results Shows that Max floc A-108 is most effective in comparison to Max floc A-107 to settling the phosphate slurry. This may be attributed due to the higher molecular weight and anionicity of A-108 than compared to P.E A-107. Dosing rate also plays very important role. P.A-108 is most effective at 6 ppm of dosages.

Influence of Feed Particle Size Distribution

1. Table-4. Clearly shows that feed at the Belt Drum Filter is predominantly consists of fine particles i.e. less than 25 microns. Finer particles have slower settling and filtration rates. The specific area is increased, which produces lower solid concentration in the thickener under flow as well as in discharge filter cake. This may result in the higher moisture content in the cake. This was confirmed by analyzing particle size distribution of feed to BDF. The sample of 09.05.05 was analyzed (see table-4) in absence of poly- electrolyte. A wet cake with a moisture content of about 20% was observed, when vacuum pressure was in the range 430-470 mm of Hg.
2. Table-7. Shows that under the similar conditioned maintained as above, a dry cake was observed at BDF-3002. In this situation P.E SS-120 and max floc A-108 were added at phosphate thickener and belt drum filter respectively. The particle size distribution was even much more finer i.e., 85 % < 25 microns. This shows that P.E dosage at multiple location plays very significant role in reducing moisture content.

CONCLUSION

- a) There is some significant influence of P.E addition at BDF distribution box but the dewatering of discharge cake is not very efficient.
- b) The dewatering of discharge cake is quite efficient in case of multiple dosing i.e. at BDF distribution box and phosphate thickener.
- c) The particle size distribution has very significant influence on the filtration .The finer size distribution(i.e., 85 % < 25 micron size of the phosphate feed) less will the chances of producing dry cake .Dewatering of concentrate cake increase with the increase in average particle size, and decrease in percentage of very fine material .
- d) Presence of P.E at multiple location increases the chances of producing dry cake provided the vacuum pressure is in the range of 430 to 450 mm of Hg.
- e) Polyelectrolyte Max flocc-A-108 is most appropriate for thickening the phosphate slurry.
- f) P.E dosage at Phosphate Thickener improves the settling of phosphate fines and decreases the turbidity of P.T over flow water.
- g) Cationic Polyacrylamide C-21 and C-33 were not found to be effective.

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