

# FLOCCULATION OF IRON ORE FINES USING MAGNAFLOC POLYMERS

K.J.Balasubramani\*, G.Bhaskar Raju\*\* and P.R.Khangaonkar\*\*\*

## ABSTRACT

Flocculation of Kudremukh Iron Ore fines was studied using two commercially available flocculants containing anionic polyacrylamide as a basic component with different molecular weight. Variables like polymer dosage on settling rate and turbidity, pH of the pulp, agitation, pulp density, effect of calcium chloride, etc. were systematically examined in and compared. Structure of the flocs, IR spectra of iron ore fines before and after flocculation were examined in order to understand the flocculation mechanism. It was found that the optimal dosage of flocculants mechanism. It was found that the optimal dosage of flocculants was around 0.05 kg/t for Anionic Magnafloc-155 and 0.07 kg/t in the case of Anionic Magnafloc-1011. It was also found that the settling characteristics are better with anionic Magnafloc-155 compared to that of Anionic Magnafloc-1011.

## INTRODUCTION

Fines and ultra fines are generated invariably during mining, milling and other metallurgical operations. Our country is on the threshold of large scale mechanised mining and processing of Iron Ores to meet both internal demands and export commitments. Huge accumulation of fines have led to environmental problems due to both dust generation and water pollution. Since the generation of fines is more on large scale operations, these fines are to be recovered and the tailings are to be discharged in an environmentally acceptable form. Presently these fines are either ignored or treated empirically by conventional mineral processing techniques.

Flocculation appears to be the most promising technique to solve these problems due to its simplicity on large scale operation and simultaneous water purification for its reuse in the process. In the past natural substances like lime and aluminous earth and natural polymers like various starches and Glue were utilised as sedimentation aids. Due to large

reagent consumption, low settling rate, high turbidity in the supernatant solution and the low shelf life, these substances were mostly replaced by synthetic water soluble polymers since 1951 [1]. Recently Magnafloc and Chemifloc series of flocculants introduced in the market have drawn the attention of many researchers due to their special properties, viz. selectivity, low reagent consumption (ppm level), low toxicity, etc. [2-4]. The present study aims to understanding the flocculation behaviour of Kudremukh Iron Ore fines using Anionic Magnafloc-155 and 1011.

## EXPERIMENTAL

### (A) Materials :

- (i) Iron Ore used in the present investigation was obtained from the mines of Kudremukh, Karnataka, and the chemical analysis of the same is shown in Table-1. The Iron Ore sample was wet-ground and subjected to sieving to obtain - 400 mesh.
- (ii) The details of the flocculants used are given in Table-2. The polymer samples were weighed accurately and added to a known volume of water and stirred to prepare the solutions of desired concentrations. Dilute sodium hydroxide and Hydrochloric acid were used to adjust the pH of the solution. Analar grade calcium chloride was used wherever necessary.

Table 1 : Chemical analysis of the iron ore

Radical	Percentage
Total Fe	66.50
SiO <sub>2</sub>	2.75
Al <sub>2</sub> O <sub>3</sub>	1.36
CaO	0.35
MgO	0.40
LOI	4.00

\* Research Fellow, \*\* Scientist, National Metallurgical Laboratory Madras Centre, CSIR Madras Complex, Madras-600113.

\*\*\* Visiting Professor, School of Mineral Resources Engineering, University Sains Malaysia, 30000, Ipoh, Perak, Malaysia.

**Table 2 : Details of the synthetic polymers**

Sl. No.	Name of the flocculant	Chemical composition	Nature of charge	Physical form
1	Magnafloc-155	Polyacrylamide based polymer	Anionic	White powder
2	Magnafloc-1011	-do-	-do-	-do-

### (B) Methods

Flocculation experiments were done in a graduated cylinder at a slurry consistency of 1% solids. Total volume of the suspension including polymer and other modifiers was made upto the mark of the cylinder by diluting with water. In each experiment, the contents of the cylinder was inverted twenty times to put the entire system under gentle shaking and was allowed to settle. The settling rate curve was monitored from the rate of descent of the mud-line (interface between suspension and the supernatant liquid) with time. The settling rate values were calculated from appropriate linear portions of the curve coinciding with uniform terminal settling velocity of the flocs and the same was expressed in cm/sec. The flocculation response of the mineral is expressed as the percentage solids settled in a fixed settling time. Turbidity measurements of the supernatant solution using Nephelo-Turbidity Meter is also followed to measure the degree of flocculation.

### RESULTS AND DISCUSSIONS

Preliminary tests were conducted to see the effect of agitation on mineral flocculation. It was observed that twenty inversions of the cylinder were enough to yield optimum flocculation. It is also observed that prolonged and vigorous agitation leads to partial redispersion and slow rate of settling.

From the Fig.1 it is clear that the steepness of Anionic Magnafloc-155 is more compared to that of Anionic Magnafloc-1011. It is well known that as the steepness of the settling curve increases, settling rate also increases and greater the steepness, larger is the relative density and size of the floc (Fig.2). The size of the floc at different flocculant dosage was

observed under a microscope. Photographs show that both the flocculants form bigger flocs with increasing concentration of the flocculants. It was also observed that Anionic Magnafloc-155 formed bigger flocs than Anionic Magnafloc-1011 (Fig. 3).

The effect of flocculant concentration on settling and turbidity of the supernatant solution at pH 4.00 is shown in Fig.4. The pH of the pulp was so chosen as to correspond to just below the isoelectric point where the Iron Ore particles are charged positively. Effective flocculation can be expected by a negatively charged anionic flocculant through charge neutralisation mechanism [5]. The dissolution of the ore was found to be negligible at this pH. It is evident from Fig.4 that the percentage of settling increases with increasing flocculant concentration. Maximum settling was noticed at 0.05 kg/t for Anionic Magnafloc-155 and 0.07 kg/t in the case of Anionic Magnafloc-1011. Beyond this concentration there was no improvement in the settling rate. However, a slight increase in turbidity was observed in supernatant solution which may be due to excess flocculant remaining unabsorbed in the solution. Hence an exact dosage of flocculant is essential to achieve the best settling characteristics. The settling is so rapid that within 10 seconds (Anionic Magnafloc-155) and 30 seconds (Anionic Magnafloc-1011) total settling of solids was noticed.

A further set of experiments was conducted to study the extent of flocculation as a function of pH (Fig.5). It was observed that the tendency of flocculation started decreasing from pH 8.00 onwards. At pH 10.00 and above, a constant rate of settling was noticed. On the other hand, as the pH is made more acidic, there is only a slight decrease in flocculation. The addition of flocculant improved the floc formation and almost complete settling was noticed within 10 seconds for Anionic Magnafloc-155 and 30 seconds in the case of Anionic Magnafloc-1011 in the acidic pH range. Excess dosage of flocculant was found to have no effect in the basis medium.

Experiments were carried out to obtain flocculation in the basis medium using these flocculants. These experiments showed that addition of calcium chloride played a significant role on the settling behaviour (Fig.6). It was found that the minimum amount of

calcium chloride required to achieve flocculation at pH 11.00 was 1.8 kg/t of Iron Ore fines and best flocculation was noticed only at 5 kg/t. The addition of calcium chloride not only improved the settling in the alkaline range but also decreased the turbidity of the supernatant solution. Fig. 7 shows the effect of pH on flocculation and calcium chloride concentrations.

The sequence of addition of flocculant and calcium chloride was found to be an important criterion. From Fig. 8, it is clear that at low concentration of flocculant, better settling and low turbidity could be obtained when calcium chloride was added first. However, when excess amount of flocculant was present, the flocculation was found to be independent of the sequence of addition. But the turbidity was found to be more when flocculant was added first. In the alkaline pH, the mineral particles acquire negative charge and there exists a strong electrical double layer repulsion between the particles and also between the partial anionic flocculant. The addition of calcium chloride maintains positive charge on the particle, thus facilitating the absorption of negatively charged flocculant. The addition of calcium chloride also reduces the electrical double layer repulsion between the particles. When an anionic flocculant was added to the pulp at this stage, flocculation was noticed and it could be due to the inter-particle bridging as reported by the earlier workers for anionic polymer flocculants [6]. It was also noticed that in the absence of the flocculant, interparticle bridging does not take place, resulting in poor or nil flocculation at this pH.

When calcium chloride was added first, flocculation occurred by the above mechanism. But when the flocculant is added first, the repulsive forces are increased since the flocculants also possess negative charge. Though the addition of calcium chloride under these conditions decreased the repulsive forces, the interparticle bridging was not so significant as noticed in the former case [6].

The flocculated bed depth obtained at different flocculant concentration was also studied and results are given in Fig.9. The bed depth was calculated after 30 minutes of the test. From the Figure it is observed that the bed depth increased initially with

flocculant concentration attaining a limiting value and thereafter there was no noticeable difference in bed depth for the two flocculants studied. The amount of flocculant absorbed on the surface of the mineral particle may be increased upto a certain extent with increasing amount of flocculant. Due to the hydration of mineral surface, it swells resulting in increased floc size. After certain addition of flocculant concentration, further absorption is not possible and the excess flocculant remains in the solution and the bed depth formed remains constant.

I.R.Spectra are recorded using KBr pellet technique to establish the mechanism of polymer absorption on mineral particles. The broad band obtained for the flocculated iron ore in the range of 3650-3400 CM confirms the hydrogen bond formation. Similar observation was made by the earlier workers for polyacrylamide-silica system [7].

The orientation of hydrogen bonding is explained with the results obtained from the flocculation response as a function of pH. If the hydrogen bonding was to be established through surface -OH groups, excellent flocculation should take place at  $\text{pH} > 6.8$  (PZC of iron ore fines), because the dissociation of carboxylate ions will be favoured with increasing pH. However in the present investigation the flocculation was found to be very poor in the basic region. This observation indirectly indicates that the hydrogen bonding is not through surface -OH groups. Since excellent flocculation is noticed in acidic pH, it is concluded that hydrogen bonding is most likely through surface oxygen species on to which the hydrogen donating groups can be attached. Also, in acid media partially hydrolyzed polyacrylamide shall have two types of hydrogen donating groups :-COOH and -CO-NH<sub>2</sub>. Here the hydrogen bond via -COOH is stronger than via -CO-NH<sub>2</sub>, due to the greater electronegativity of the oxygen atoms.

## CONCLUSION

The following conclusions were drawn from the this study :

1. Both Anionic Magnafloc-155 and 1011 could be used as flocculants for Kudremukh Iron Ore fines and that the use of both of them resulted in rapid and complete settling. Amongst the two floccul-

ants studied, Anionic Magnafloc -155 was found to result in better settling characteristics. The optimum dosages were 0.05 kg/t and 0.07 kg/t for Anionic Magnafloc-155 and 1011 respectively.

2. Addition of calcium chloride improved both settling rate and clarity of the supernatant solution significantly in alkaline pH range whilst in acidic pH range, there was no need for addition of calcium chloride to achieve the desired flocculation.
3. The sequence in which calcium chloride and the flocculant added to the pulp was an important factor in deciding the efficiency of flocculation.
4. The size and shape of the floc was found to vary with the flocculant concentration and the nature of the flocculant.
5. From the experiment results, it was concluded that effective flocculation was due to charge neutralisation and inter-particle bridging mechanisms.

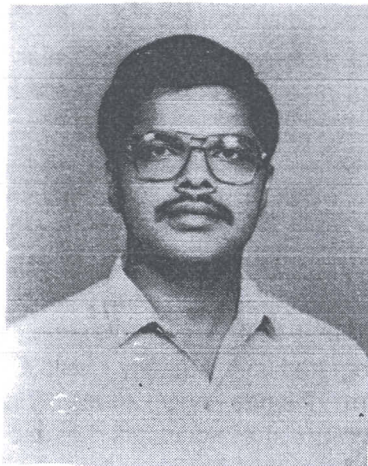
#### ACKNOWLEDGEMENT

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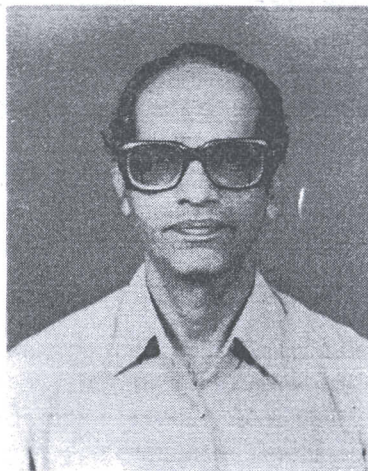
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#### BIO DATA OF AUTHORS



**Dr. G.Bhaskar Raju** is presently working as a Scientist at NML Madras Centre. Post graduated in chemistry from S.V.University, Tirupathi and obtained doctorate degree from the same University on the subject of "Studies on the flotation of fine particles-electroflotation of chalcopyrite fines". He has got 10 years of R&D experience in the field of mineral processing and around 12 publications for his credit.

**K.J.Balasubramani** post graduated in Analytical Chemistry from the University of Madras and presently working as a research fellow at NML Madras Centre in the field of mineral processing especially on the beneficiation of mineral fines by flocculation



**Professor P.R.Khangaonkar** Born in 1935, he obtained his B.E. (Mech.) in 1956, BE (Met.) in 1957 and ME (Met.) in 1959 from the College of Engineering, Pune. He obtained Ph.D. (Met.) from Rensselaer Polytechnic Inst, Troy, USA in 1962. Dr. P.R.Khangaonkar has worked in various capacities in REC, Nagpur and NML Madras Centre and has published 3 books and more than 75 papers in leading journals. He has guided research fellows and produced 9 Ph.Ds so far. Presently he is working as Visiting Professor, School of Mineral Resources Engineering, University Sains Malaysia.

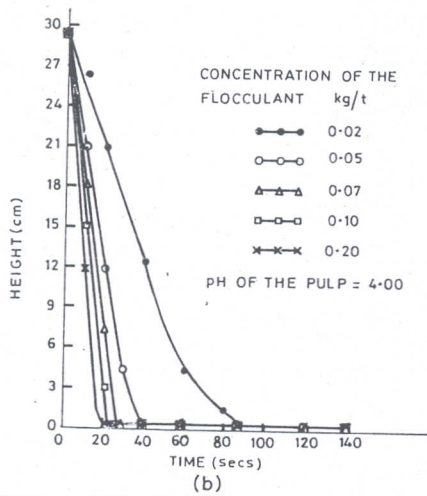
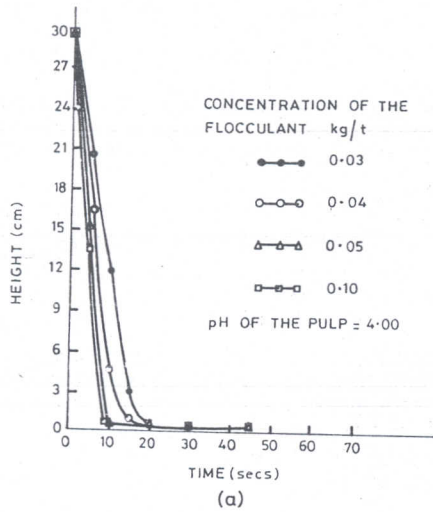


FIG.1 TYPICAL SETTLING CURVES OF IRON ORE FINES WITH  
(a) ANIONIC MAGNA FLOC-155 (b) ANIONIC MAGNA FLOC-1011

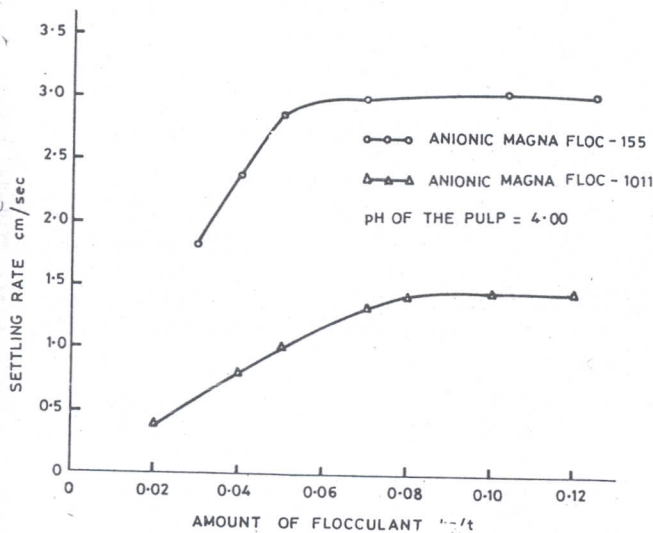


FIG.2 SETTLING RATE OF IRON ORE FINES AS A FUNCTION OF FLOCCULANT CONCENTRATION

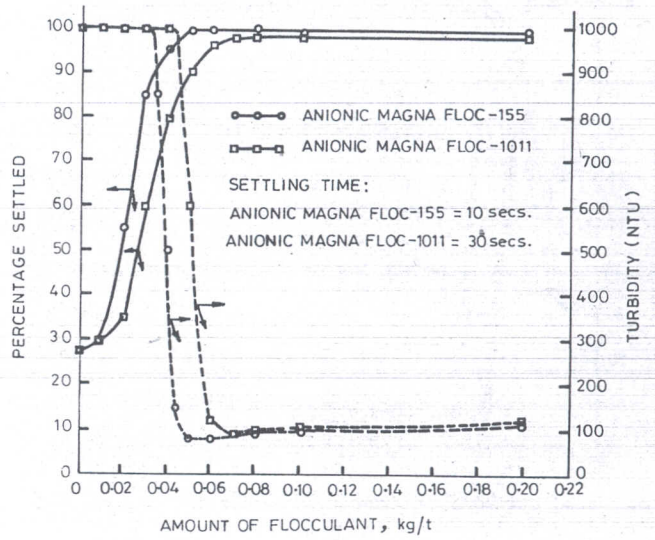


FIG.4 PERCENTAGE SETTLED AND TURBIDITY OF THE SUPERNATANT AS A FUNCTION OF FLOCCULANT CONCENTRATION AT pH 4.0

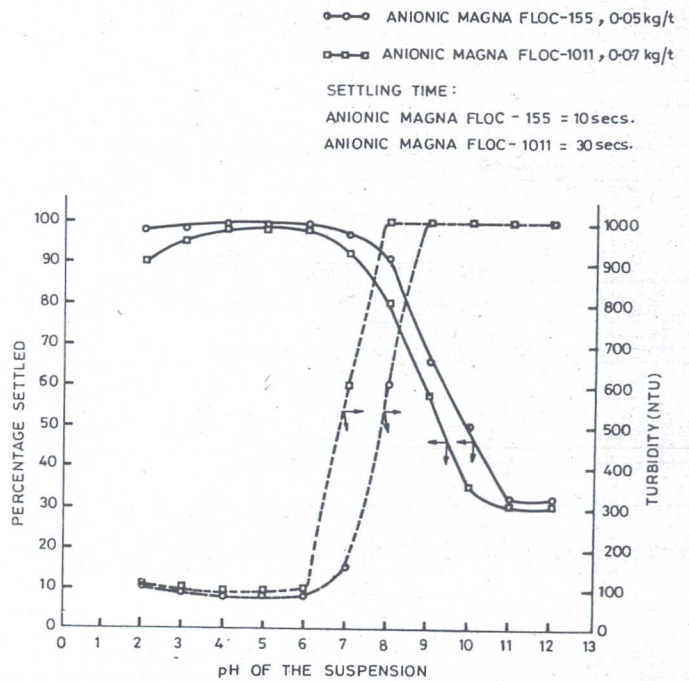


FIG.5 PERCENTAGE SETTLED AND TURBIDITY OF THE SUPERNATANT AS A FUNCTION OF pH

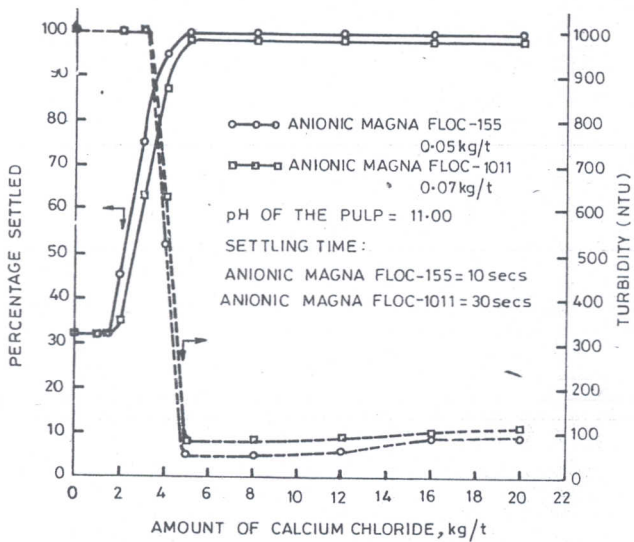


FIG.6 EFFECT OF CALCIUM CHLORIDE ON FLOCCULATION OF IRON ORE FINES

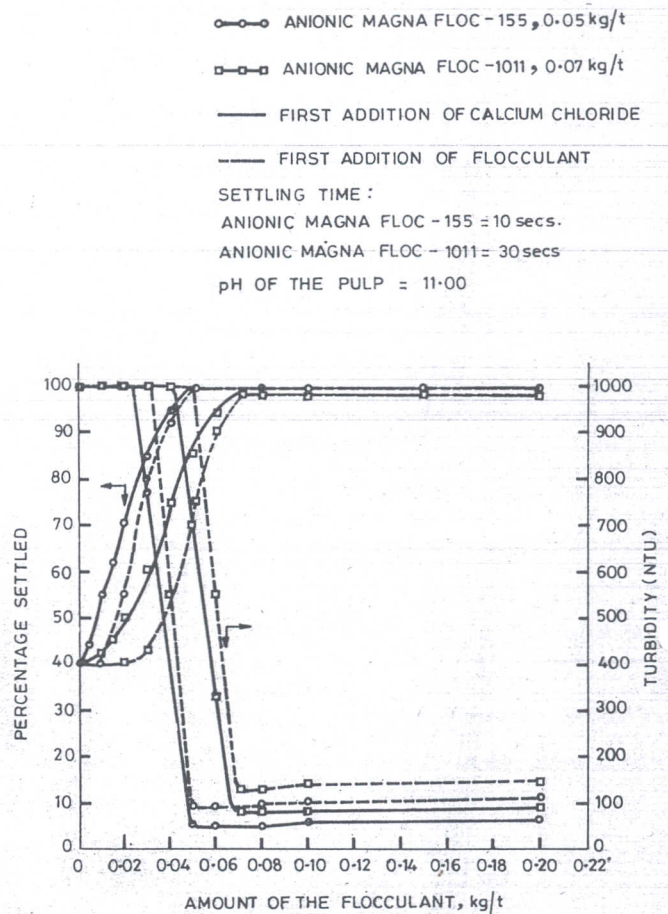


FIG.8 EFFECT OF THE SEQUENCE OF ADDITION OF FLOCCULANT AND CALCIUM CHLORIDE

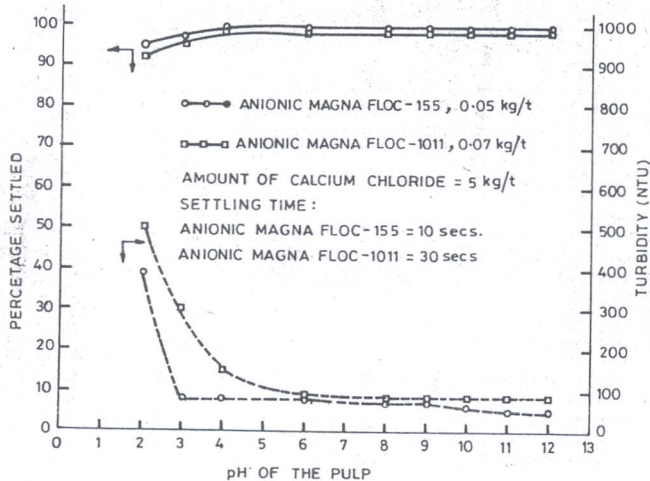


FIG.7 EFFECT OF pH ON FLOCCULATION AT FIXED CALCIUM CHLORIDE AND FLOCCULANT CONCENTRATION

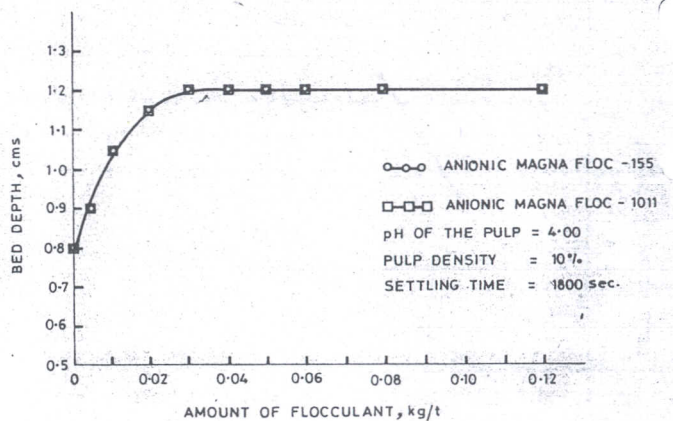


FIG.9 VARIATION OF FLOCCULATED BED DEPTH WITH FLOCCULANT CONCENTRATION

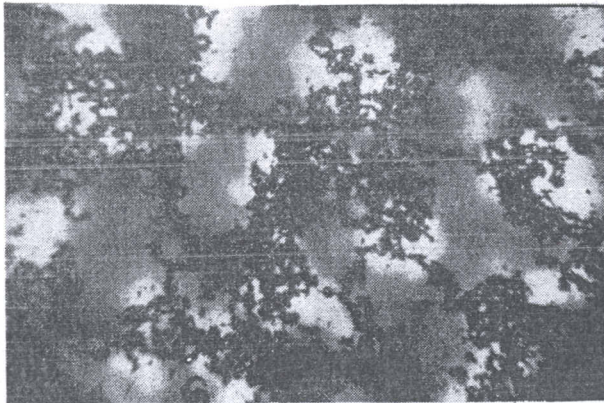
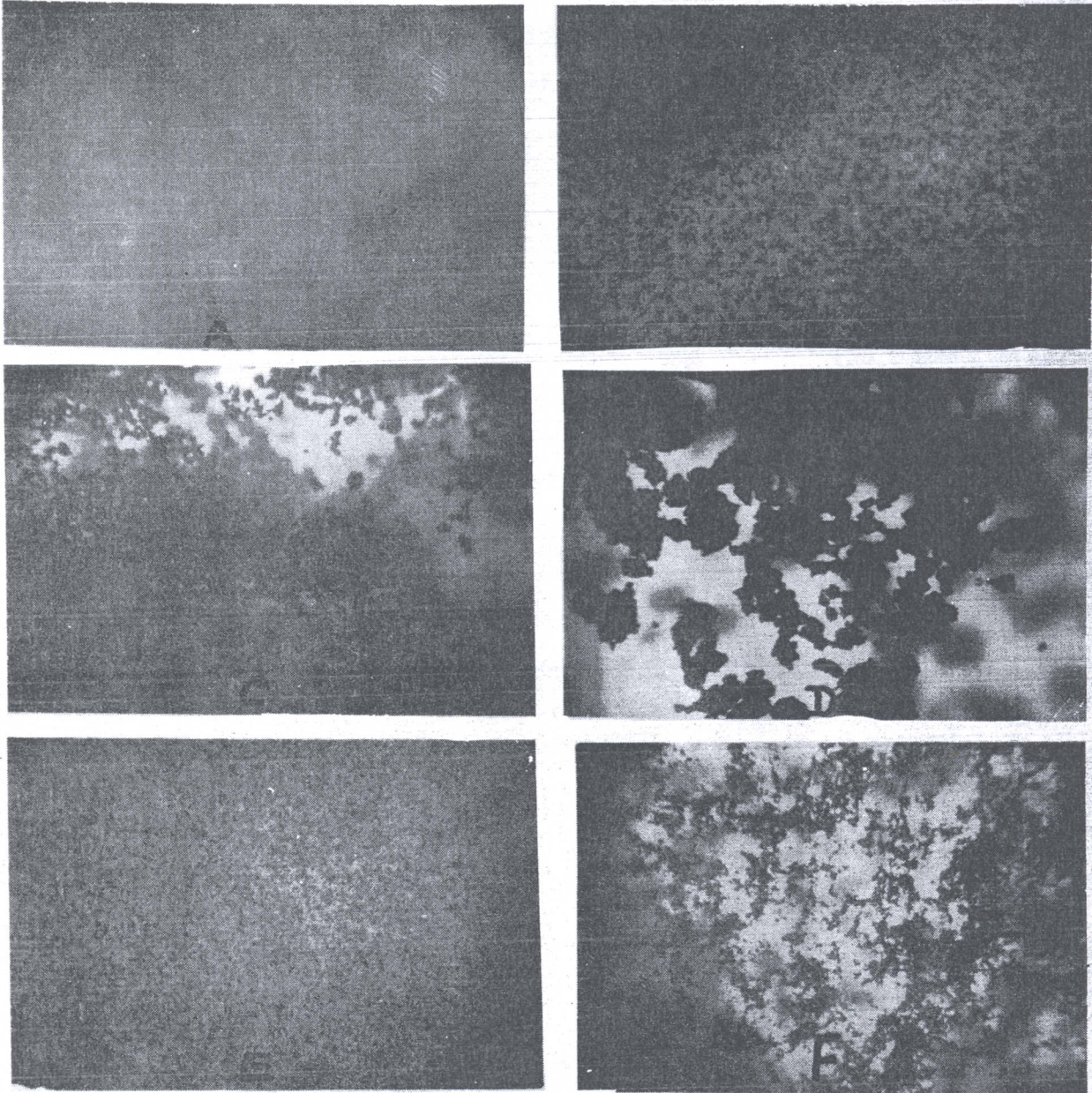


Fig. 3: Effect of flocculant concentration on floc size (X20) at pH 4.00

- a) Suspension of iron ore fines before flocculation
- (b) Flocculated iron ore using anionic magnafloc-155 of 0.025 kg/t (less concentration)
- (c) Flocculated iron ore using anionic magnafloc-155 of 0.05 kg/t (optimum concentration)
- (d) Flocculated iron ore using anionic magnafloc-155 of 0.1 kg/t (excess concentration)
- (e) Flocculated iron ore using anionic magnafloc-1011 of 0.025 kg/t (less concentration)
- (f) Flocculated iron ore using anionic magnafloc-1011 of 0.07 kg/t (optimum concentration)
- (g) Flocculated iron ore using anionic magnafloc-1011 of 0.1 kg/t (excess concentration)