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SEPARATION OF CARBON FROM DEINKING WASTE  
BY MEANS OF FLOTATION

A Senior Thesis

Conducted under the supervision of Mr.  
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Submitted June 6, 1955

*Lester R. Beeman*

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## ABSTRACT

The technique of froth flotation, as practiced in the separation of mineral ores, has been applied to the separation of carbon black from the deinking waste of the paper industry.

On the theory that carbon black (from ink) has a positive charge in water, and clay and cellulose fibers have negative charges in water, experiments were of two types. First, it was attempted to float the carbon from the clay and cellulose fibers. Second, it was attempted to float the clay and cellulose fibers from the carbon.

Several possible formulas were found for separating carbon black from deinking waste. Favorable results were obtained in both types of experiments.

## LITERATURE SURVEY

### Introduction

In the production of deinked paper stock from waste paper for the making of fine papers, there is created a large tonnage of deinking waste. The purpose of this work is to devise a method of separating carbon black, derived from printing ink, from deinking waste by means of flotation. There is need for such a method not only from the standpoint of reducing the amount of waste material from the deinking industry, but also for the recovery of valuable cellulose fibers and clay fillers.

### Principle of Flotation

A survey of the literature showed that there is no previous published material on the flotation of deinking waste, although there has been some work done in separating ink from paper, therefore this survey will deal primarily with flotation in general.

Flotation refers specifically to the art and science of separating solid particles from each other in a liquid pulp, or suspension, by means of air bubbles. In froth flotation (by far the most usual form as opposed to bulk oil flotation and skin flotation) a froth is formed by introducing air into a pulp which consists of the finely divided particles and water, to which a frothing agent has been added. Those minerals that have an affinity for air bubbles rise to the surface in

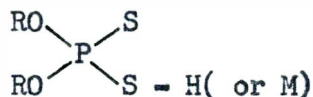
the froth and are thus removed from those that are completely wetted.

## Chemical Factors

### Collectors

In principle, flotation depends on the relative wettability of surfaces (1). A flotation system consists of a liquid, a gas, and some particulate material. The surface of the material may exhibit a natural nonwettability, but it usually must be treated with various flotation reagents in order to produce the desired degree of wettability or nonwettability. These reagents are called "collectors". They are divided into two classes -- anionic and cationic (2). The anionic class is selective to those minerals which have a high concentration of positive surface charges. The cationic class is selective to those minerals which have a high concentration of negative surface charges.

Anionic collectors are used in the great majority of present day ore flotation operations. They are grouped into classes based on the active or polar groups (1), the most common of which are sulfhydryl (mercapto) or carboxylic. The sulfhydryl class includes types such as xanthates and thiophosphates with the general structural formulas:



R = alkyl group      M = alkali metal or ammonium ion

The carboxylic collectors are principally fatty acids or their soaps.

Cationic collectors include quaternary ammonium, pyridinium, quinolinium or sulfonium salts and amine derivatives of fatty acids. The amines are the most versatile of all known reagents for non-metallic mineral separations (2). The development of these cationic reagents during the last 15 to 20 years has been the major factor in the advance of the field of non-metallic mineral separations.

Every collector should possess the following characteristics (2):

- a) It must possess a greater affinity for the particle that is being concentrated than for the other particle constituents of the pulp.
- b) It must be relatively or completely soluble in water, or compatible with a particle-water pulp.
- c) It should be non-corrosive.
- d) It should be quite stable chemically.



- e) It must be a potent collector which has the ability to promote speedy and rapid flotation of particles with a minimum of contact time preceding flotation.

The polar end of the collector attaches itself to the mineral surface either by chemical or physical adsorption; the nonpolar unreactive end is oriented away from the mineral surface. The result is a hydrophobic coating at the mineral surface which permits attachment to the air bubbles. It is this surface condition which permits the floating of minerals which normally sink in water.

Quite often both cationic and anionic collections are used in consecutive circuits under entirely different conditions to produce a satisfactory process.

### Modifiers

If other ions are present in solution, in addition to the hydrogen and hydroxyl ions, they exert profound effects on the attachment of collector ions to the mineral surfaces and thus give rise to a large class of so-called "modifying reagents" (1). Modifying agents may be classed in one of the following groups, according to their functions (3):

pH control  
Depressant  
Activator  
Sulfidizer  
Disperser or deflocculator  
Protective colloid

Examples of pH control agents are lime, soda ash, and sulphuric acid. Where it is desirable to carry out flotation on the alkaline side and lime can be used, this low cost material is preferred. Soda ash is usually used where lime may be objectionable. Sulfuric acid is the usual agent for control of pH on the acid side.

Lime, sodium cyanide, and certain dichromates are examples of depressants. These reagents prevent the attachment of the collecting ion to the particle surface. Depressants are necessary where it is desirable to prevent flotation of one mineral normally floatable along with another being removed.

Certain modifying agents, termed activators, serve to bring about the attachment of collectors to particles by means of an ionic bridge. When a material has been depressed or is not normally floatable an activator is used to bring about its active flotation.

Sodium Sulfide is the common sulfidizing agent. Sulfides are easily floated with xanthate type collectors.

Gangue, at times, may be of such a nature as to contain materials having a tendency to flocculate or clump, or hold together to such an extent as to interfere with efficient flotation of the desired material. Then, it becomes necessary to apply deflocculators and dispersants, and protective colloids are then used to hold them in the dispersed state. Sodium silicate is used as a dispersant. Starch, Casein, and glue are examples of materials used to disperse both gangue and carbonaceous materials and to serve as protective colloids to prevent reflocculation.

Since the reactions under consideration take place at the surfaces of minerals, and also since only a partial monomolecular layer is required to produce flotation in most cases, the quantity of dissolved ion necessary to affect the reactions is very small. No fixed rule can be given to the amounts of flotation agents to be used. The optimum quantity varies with the material being beneficiated.

#### Physical Factors

Once the particle has been prepared with a water-repellent surface, it must be brought into contact with an air-

water interface in such a manner that it will remain at that interface long enough for a separation to be effected. The most common method of producing these conditions is by the use of froth flotation, in which air bubbles are introduced into a pulp in any of a variety of ways.

A good flotation bubble must be small, and it must not join the other bubbles until it gets to the top of the slurry (4). Flotation engineers have determined that bubble structure is an important factor in flotation recovery (5). Experience proves that it is the surface area -- not the size of the bubbles -- which controls recovery. Small bubbles are desirable because they are more active and expose a greater surface area.

### Frothers

When the coated bubbles rise to the surface, they must remain stable a sufficient time for the froth to be removed. To produce a froth that will be persistent enough to allow a separation, a number of frothing agents may be used. Frothers are chemicals that ionize to a very slight extent in water and only very slightly (4). One ion must be hydroxyl, as bubbles in water have their surfaces composed in part from these hydroxyl ions. The mating ion forming the rest of the

frother should be a chemical chain, generally a carbon hydrogen chain. The most commonly used frother is pine oil (an impure terpineol  $C_{10}H_{17}OH$ ). Amyl alcohol, other long chain alcohols, phenol, cresylic acid, creosote, and others are also used. Aliphatic alcohols containing five to ten carbon atoms are frothers which have more recently found application in this field. Frothers function by changing the surface tension of the liquid (1). The froth serves a secondary purpose in providing a zone in which the unfloated mineral particles (pulp that has been mechanically entrapped) will drain away from the flotation concentrate.

Excess frother will reduce the selectivity by producing too much froth, or, in extreme cases, will destroy the froth completely (1).

### Conditioning

Most minerals can be floated from their ores quickly after addition of the flotation agents to the pulp. However, some ores require conditioning before flotation takes place. In some cases, this conditioning is simply a matter of allowing longer contact time between flotation agents and mineral particles before flotation sets in. In other cases, the pulp must be given a preflotation attrition before the surface of

the mineral particle can accept the collector agent (3).

#### Particle size

The particle diameter of carbon black (the average diameter) probably does not exceed 0.1 micron (8), while clays range in particle diameter from 0.05 micron to 20 microns (9). Coating clays contain from 70 to 95% particles which are finer than 2 microns (10).

Fine particles in the near-colloidal size range are difficult to recover by flotation. This is due in part to the mechanical difficulty of bringing fine particles into contact with air bubbles (6) and in part to the fact that the surfaces of fine particles are not the same as a freshly produced surface. Gaudin and Malozemoff (7) postulate that fine particles have a surface older on the average than course particles. Thus, some ores require a scrubbing or attrition treatment to expose fresh mineral particle surfaces to the collector agent.

#### Water Supply

Water supplies in adequate quantity and purity are necessary.

## Equipment

Froth flotation cells are classified in accordance with the mode of introduction of the gas as: (a) agitation cells in which air is drawn down by a vortex caused by a rotating impeller; (b) subaeration cells in which air is introduced by suction or by blowing through or to the base of a rotating impeller; (c) cascade cells in which air is introduced by tumbling of the pulp; and (d) pneumatic cells in which air is introduced directly by blowing.

In agitation cells the design and speed of the impeller precisely controls the number and size of the bubbles (4).

## Variables

The common variables during operation are (1):

Height of the pulp in the cell

Degree of aeration

Pulp density

Temperature of the pulp

A relatively lower depth of pulp means a deeper froth layer and usually results in a cleaner overflow.

A more intense aeration may provide a greater probability of particle-bubble contact, but may also result in a concentrate containing more undesirable impurities.

Pulp density is important in both chemical and mechanical considerations. It determines reaction time and the quantity of reagent required to give the desired reagent concentrations; hence, chemical factors argue for higher pulp densities. On the other hand, mechanical factors place definite limits on the optimum density. Flotation is normally more rapid and complete in denser pulps, but concentrate grade is lower; however there are some exceptions to this rule (11).

Temperature of the pulp has an effect on the rate of reagent reaction. Usually, flotation is carried out at the temperature assumed by the pulp. In some cases, however, it is necessary to raise the temperature of the pulp in order to accelerate the slow action of the collector at the normal temperature.



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## EXPERIMENTAL OUTLINE

### Procedure

Inasmuch as carbon black has a positive (+) charge and clay and cellulose fibers have a negative (-) charge, this experimental work will be carried out in two parts. First, it will be attempted to float the carbon from the clay and cellulose fibers with anionic type collectors; secondly, it will be attempted to float the clay and cellulose fibers from the carbon with cationic type collectors.

In order to judge the amount of separation of carbon from the deinking waste, brightness standards will be set up using HUBER clay (a predispersed Georgia clay) and carbon black. These standards will consist of the following percentages of clay and carbon black:

<u>% Clay</u>	<u>% Carbon Black</u>
100.0	0
99.75	.25
99.5	.5
99.25	.75
99.0	1.0
98.5	1.5
98.0	2.0

The brightness of the deinking waste will be taken before and after flotation. In this way a comparison will be made as to how much carbon has been removed from the deinking waste. Brightness samples of the original deinking waste will be collected on filter paper by means of a small buchner funnel. These samples will then be dried at 105° C to a constant weight. Brightness samples of the

floated deinking waste will be obtained by removing a small portion of the clay-cellulose fraction from the flotation machine. These samples will then be filtered and dried in the same manner as the original deinking waste.

All brightnesses will be taken with the Photovolt brightness tester.

Any system which shows promise of a good separation in a single stage operation will be assumed to be improved by a multiple stage operation. If time permits a multiple stage operation will be run. Also, any experiment which clearly indicates that a later experiment will give the same results will be sufficient reason for omitting the later experiment.

#### Equipment

The equipment used in these experiments will consist of a laboratory size Fagergren Flotation Machine of the batch operated type. The flotation chamber is of 600 gram capacity and air is introduced by direct agitation. An impeller speed of 1800 RPM will be used in all experiments.

## Part I

### Flotation of Carbon from Clay and Cellulose

#### Chemicals

##### Collectors:

AERO Promoter 708 - fatty acid

AERO Promoter 710 - sodium soap of AERO Promoter 708

Oleic acid - unsaturated fatty acid

##### Frothers:

Pine Oil - an impure terpeneol

AEROFROTH 63 - higher aliphatic alcohols

AEROFROTH 65 - water soluble synthetic frother

AEROFROTH 80 - long chain alcohol

##### Depressants:

Sodium Silicate

Table I Experiments

Exp.	Collector	Amount lb/ton	Frother	Amount lb/ton	Depressant	Amount lb/ton
1	AERO Promoter 708	0.5	Pine Oil	0.1	----	
2	" "	" "	" "	"	Sodium Silicate	1.7
3	" "	" "	AEROFROTH 63	0.25	----	
4	" "	" "	" "	"	Sodium Silicate	1.7
5	" "	" "	AEROFROTH 65	0.25	----	
6	" "	" "	" "	"	Sodium Silicate	1.7
7	" "	" "	AEROFROTH 80	0.1	----	
8	" "	" "	" "	"	Sodium Silicate	1.7

Table I — continued

Exp.	Collector	Amount lb/ton	Frother	Amount lb/ton	Depressant	Amount lb/ton
9	AERO Promoter 710	0.5	Pine Oil	0.1	----	
10	" "	"	" "	"	Sodium Silicate	1.7
11	" "	"	AEROFROTH 63	0.25	----	
12	" "	"	" "	"	Sodium Silicate	1.7
13	" "	"	AEROFROTH 65	0.25	----	
14	" "	"	" "	"	Sodium Silicate	1.7
15	" "	"	AEROFROTH 80	0.1	----	
16	" "	"	" "	"	Sodium Silicate	1.7
17	Oleic Acid	0.5	Pine Oil	0.1	----	
18	" "	"	" "	"	Sodium Silicate	1.7
19	" "	"	AEROFROTH 63	0.25	----	
20	" "	"	" "	"	Sodium Silicate	1.7
21	" "	"	AEROFROTH 65	0.25	----	
22	" "	"	" "	"	Sodium Silicate	1.7
23	" "	"	AEROFROTH 80	0.1	----	
24	" "	"	" "	"	Sodium Silicate	1.7

## Part II

## Flotation of Clay and Cellulose from Carbon

Collectors:

AEROMINE 2026

ARMAC - coco amine

DUOMAC S - tallow diamine

Frothers:

Pine Oil - an impure terpeneol

AEROFROTH 63 - higher aliphatic alcohols

AEROFROTH 65 - water soluble synthetic frother

AEROFROTH 80 - long chain alcohol

Depressants:

Starch

AERO Depressant 645

Collector Assisting Agents:

Fuel Oil

Table II Experiments

Exp.	Collector	Amount lb/ton	Frother	Amount lb/ton	Depressant	Amount lb/ton	Coll't Ast/ag't	Amount lb/ton
1	AEROMINE 2026	0.25	Pine Oil	0.1	Starch	0.5	----	
2	" "	"	" "	"	"	"	Fuel Oil	0.5
3	" "	"	" "	"	AERO Depr. 645	0.5	----	
4	" "	"	" "	"	" " "	"	Fuel Oil	0.5
5	" "	"	AEROFROTH 63	0.25	Starch	0.5	----	
6	" "	"	" "	"	"	"	Fuel Oil	0.5
7	" "	"	" "	"	AERO Depr. 645	0.5	----	
8	" "	"	" "	"	" " "	"	Fuel Oil	0.5
9	" "	"	AEROFROTH 65	0.25	Starch	0.5	----	
10	" "	"	" "	"	"	"	Fuel Oil	0.5
11	" "	"	" "	"	AERO Depr. 645	0.5	----	
12	" "	"	" "	"	" " "	"	Fuel Oil	0.5
13	" "	"	AEROFROTH 80	0.1	Starch	0.5	----	
14	" "	"	" "	"	"	"	Fuel Oil	0.5
15	" "	"	" "	"	AERO Depr. 645	0.5	----	
16	" "	"	" "	"	" " "	"	Fuel Oil	0.5
17	ARMAC C	0.25	Pine Oil	0.1	Starch	0.5	----	
18	" "	"	" "	"	"	"	Fuel Oil	0.5
19	" "	"	" "	"	AERO Depr. 645	0.5	----	
20	" "	"	" "	"	" " "	"	Fuel Oil	0.5
21	" "	"	AEROFROTH 63	0.25	Starch	0.5	----	
22	" "	"	" "	"	"	"	Fuel Oil	0.5
23	" "	"	" "	"	AERO Depr. 645	0.5	----	
24	" "	"	" "	"	" " "	"	Fuel Oil	0.5
25	" "	"	AEROFROTH 65	0.25	Starch	0.5	----	
26	" "	"	" "	"	"	"	Fuel Oil	0.5
27	" "	"	" "	"	AERO Depr. 645	0.5	----	
28	" "	"	" "	"	" " "	"	Fuel Oil	0.5

Table II — Continued

Exp.	Collector	Amount lb/ton	Frother	Amount lb/ton	Depressant	Amount lb/ton	Coll't Ast/ag't	Amount lb/ton
29	ARMAC C	0.25	AEROFROTH 80	0.1	Starch	0.5	----	
30	" "	"	"	"	"	"	Fuel Oil	0.5
31	" "	"	"	"	AERO Depr. 645	0.5	----	
32	" "	"	"	"	" " "	"	Fuel Oil	0.5
33	DUOMAC S	0.25	Pine Oil	0.1	Starch	0.5	----	
34	" "	"	" "	"	"	"	Fuel Oil	0.5
35	" "	"	" "	"	AERO Depr. 645	0.5	----	
36	" "	"	" "	"	" " "	"	Fuel Oil	0.5
37	" "	"	AEROFROTH 63	0.25	Starch	0.5	----	
38	" "	"	"	"	"	"	Fuel Oil	0.5
39	" "	"	"	"	AERO Depr. 645	0.5	----	
40	" "	"	"	"	" " "	"	Fuel Oil	0.5
41	" "	"	AEROFROTH 65	0.25	Starch	0.5	----	
42	" "	"	"	"	"	"	Fuel Oil	0.5
43	" "	"	"	"	AERO Depr. 645	0.5	----	
44	" "	"	"	"	" " "	"	Fuel Oil	0.5
45	" "	"	AEROFROTH 80	0.1	Starch	0.5	----	
46	" "	"	"	"	"	"	Fuel Oil	0.5
47	" "	"	"	"	AERO Depr. 645	0.5	----	
48	" "	"	"	"	" " "	"	Fuel Oil	0.5

## DATA

The experiments in this study were carried out as planned in the experimental outline (see page 14). Samples of both the original deinking waste and the clay-cellulose fraction of the floated waste were collected on filter paper by means of a Büchner funnel. These samples were dried at 105° C, and brightness readings were taken with a Photovolt brightness tester. In this way a comparison was made as to how much separation had taken place between the carbon black and the clay-cellulose fractions.

Comparative brightness measurements, made up from a commercial coating clay and carbon black, showed that very little carbon black is needed to depress the brightness of coating clay. This would also be true of deinking waste. Addition of 0.1% by weight of carbon black lowered the brightness of the clay from 84% to 61%; 0.5% lowered the brightness from 84% to 27%; only 2.0% was required to lower the brightness from 84% to 0%.

Brightness readings on the clay-cellulose fractions of the deinking waste, however, did not give adequate results as to how much separation had taken place. This was due to the fact that the clay-cellulose fractions were yellow in color. It was decided, therefore, to use percentage of reflectance



with an amber filter attachment on the Photovolt cell as a means of comparison. The results of these readings and the brightness readings are shown in tables III and IV on pages 22 and 23.

All experiments were carried out at a pH of 6.7 to 7.0 and a consistency of 1.5 to 2.5%, by weight, of deinking waste. The temperature was that assumed by the pulp. The pulp was stirred for five minutes after addition of the flotation reagents before air was introduced, and the flotation time varied from five to fifteen minutes. An excess of flotation reagents (0.1 to 0.5 grams) was used in each case to ensure maximum results.

Ash tests run on the clay-cellulose fractions after flotation showed ash contents from 7 to 15%, compared to approximately 50% on the original deinking waste. This would indicate that a larger portion of the cellulose was recovered than of the clay. It is believed that the relatively small size of the carbon black particles and the clay particles would cause this. According to the literature on ore flotation, selectivity between particles is greatly reduced when particle size is below 20 microns.

TABLE III

Flotation of Carbon from Clay and Cellulose  
(see experiments on page 16)

Exp.	% Brightness Before Flotation	% Brightness After Flotation	Diff.	% Reflectance Before Flotation (yellow filter)	% Reflectance After Flotation (yellow filter)	Diff.
1	38	42	4	40	53	13
2	"	41	3	"	51	11
3	"	44	6	"	57	17
4	"	42	4	"	51	11
5	"	42	4	"	49	9
6	"	40.5	2.5	"	47	7
7	"	42.5	4.5	"	52	12
8	"	42	4	"	51	11
9	42.5	44	1.5	47	51	4
10	"	44	1.5	"	52	5
11	"	43.5	1	"	52	5
12	"	46	3.5	"	55	8
13	"	43	.5	"	51	4
14	"	43	.5	"	52	5
15	"	41.5	-1	"	48	1
16	"	43	.5	"	51	4
17	"	39	-3.5	"	46	-1
18	"	44	1.5	"	52	5
19	"	45	2.5	"	53.5	6.5
20	"	46	3.5	"	54	7
21	"	44	1.5	"	50	3
22	"	42	-.5	"	49	2
23	42	45	3	46	53	6
24	"	39	-3	"	43	-3

TABLE IV

Flotation of Clay and Cellulose from Carbon  
(see experiments of page 17)

Exp.	% Brightness Before Flotation	% Brightness After Flotation	Diff.	% Reflectance Before Flotation (yellow filter)	% Reflectance After Flotation (yellow filter)	Diff.
1	42	40	- 2	46	56	10
2	"	44	2	"	59	13
3	"	39	- 3	"	47	1
4	"	40	- 2	"	48	2
5	"	43	1	"	56	10
6	"	43	1	"	57	11
7	"	37.5	- 4.5	"	45	- 1
8	"	36	- 6	"	41	- 5
9	"	42	0	"	57	11
10	"	42	0	"	55	9
11	"	32	-10	"	30	-16
12	"	39	- 3	"	48	2
13	"	43.5	1.5	"	53	7
14	43	44	1	45	57	12
15	"	39	- 4	"	46	1
16	"	38	- 5	"	42	- 3
17	"	42.5	- .5	"	52	7
18	"	43	0	"	49	4
19	"	38.5	- 4.5	"	44	- 1
20	"	37	- 6	"	40	- 4
21	"	41	- 2	"	56	11
22	"	43	0	"	54	9
23	"	41	- 2	"	52	7
24	"	41	- 2	"	52	7
25	"	41	- 2	"	56	11
26	"	42	- 1	"	55	10
27	"	40	- 3	"	48.5	3.5
28	"	39	- 4	"	48	3
29	"	41.5	- 1.5	"	52.5	7.5
30	"	41.5	- 1.5	"	54	9
31	"	40	- 3	"	50	5
32	"	40	- 3	"	47	2
33	"	40	- 3	"	51	6
34	"	41	- 2	"	50	5
35	"	37	- 6	"	41	- 4
36	"	38	- 5	"	42	- 3

TABLE IV — Continued

Exp.	% Brightness Before Flotation	% Brightness After Flotation	Diff.	% Reflectance Before Flotation (yellow filter)	% Reflectance After Flotation (yellow filter)	Diff.
37	39	42	3	42	56	14
38	"	42	3	"	57	15
39	"	37.5	- 1.5	"	42	0
40	"	40	1	"	47	5
41	"	41	2	"	52	10
42	"	40	1	"	55	13
43	"	38	- 1	"	42	0
44	"	37	- 2	"	43.5	1.5
45	"	41	2	"	51	9
46	"	42	3	"	52	10
47	"	38	- 1	"	42	0
48	"	40	1	"	45.5	3.5

#### Summary

The experiments were divided into two categories. First, it was attempted to float the carbon black from the clay and cellulose. Second, it was attempted to float the clay and cellulose from the carbon black.

The experiments showed a wide range of differences in the reflectance readings taken on the clay-cellulose fraction after flotation. They ranged from a difference of -16 to +17 compared to the original samples. The low negative readings obtained were caused by a bluish-black carbon depressant which was partially retained in the clay-cellulose fraction.

In the separation of carbon black from the clay and cellulose, two different floats showed promise, No. 1 and No. 3. No. 1 showed a gain on the reflectance meter of 13 points. No. 3 showed a gain of 17 points.

In the separation of clay and cellulose from carbon black, four different floats showed promise, Nos. 2, 37, 38, and 42. No. 2 showed a gain on the reflectance meter of 13 points, No. 37 a gain of 14 points, No. 38 a gain of 15 points, and No. 42 a gain of 13 points.

#### Conclusions

On the basis of these experiments it is believed that a definite and useful separation of carbon black from deinking waste can be made. This separation would allow the recovery of cellulose fibers and clay fillers.

#### Recommendations For Further Work

It is felt that additional flotation experiments would have to be carried out before this type of recovery could approach commercial application. Many variables, which were held constant in this work, such as temperature, pulp density, pH of the pulp, and optimum reagent amounts, should be investigated.

End