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Chapter 19

THE ROLE OF NONPOLAR OILS AS FLOTATION REAGENTS

by

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ABSTRACT

Nonpolar hydrocarbon oils are commonly used in the flotation of minerals to fulfill a number of roles. These roles include reducing overall reagent costs by replacing part of the collector addition, improving coarse particle recovery without reducing selectivity, as well as several others. This paper will cover the types of oils which are available, their physical and chemical characteristics, and the effect of these characteristics on the function of hydrocarbon oils in flotation. In addition, techniques for improving the behavior of oils will be discussed.

INTRODUCTION

Nonpolar oils are used as reagents in the flotation of many different types of minerals. Some typical examples are given in Table 1. The mechanism whereby a nonpolar oil acts is to coat a weakly or strongly hydrophobic particle following which it improves the strength of adhesion of the air bubble-particle contact. The means by which a particle has achieved hydrophobicity is of minor importance; whether it is natural hydrophobicity, as in the case of coal or molybdenite, or induced hydrophobicity, as in the case of silicate or sulfide flotation. From this discussion, it is apparent that the function of a nonpolar oil is controlled by its break-up and dispersion as drops throughout the slurry, oil drop-particle interaction leading to collision, and oil coating of the particle's surface. In turn, these factors are controlled by interaction between the physical environment established in a conditioner or flotation cell by the hydrodynamic flow field, the chemical environment

established by organic and inorganic, ionic and nonionic species in the process water, and the physical-chemical characteristics of the oil.

Table 1. Typical minerals recovered using reagent regimes including nonpolar oils

Type of Mineral	<u>Mineral</u>				
Naturally hydrophobic	Coal, graphite, sulfur, molybdenite				
Weakly hydrophobic	-				
sulfides	Chalcopyrite				
Hydrophilic silicates	Mica, feldspar, spodumene				
Hydrophilic oxides	Hematite, ilmenite				
the decombest to a sect at a con-	TIMENICE				
Hydrophilic oxidized					
sulfides	Malachite				
Hydrophilic salt type	***************************************				
minerals	Phosphates				

NONPOLAR OIL CHEMISTRY

The chief source of nonpolar oil collectors is from the fractional distillation of petroleum (crude oil), which is a nonuniform, highly complex mixture of hydrocarbons and hydrocarbon derivatives ranging in molecular weight from methane to heavy bitumen. Small amounts of sulfur containing and even smaller amounts of nitrogen, oxygen, vanadium, nickel, and arsenic containing compounds are usually present. The general bulk chemical analysis of crude oil falls into the range of: 83-87% C, 11-15% H, 0-6% S, 0-0.5% N and 0-3.5% O. The exact composition depends on the source of the crude. Sour crudes contain odorous sulfur compounds, usually in the

form of mercaptans or H2S. Sweet crudes contain very little sulfur.

The hydrocarbons present in crude oil may be divided into the following general chemical classes:

- Open chain (or aliphatic) compounds:
 - a. n-paraffin series, C_nH_{2n+2} . These compounds make up a larger fraction of most crudes than any other individual class. Important members are n-hexane and n-heptane.
 - Iso-paraffin series, C_nH_{2n+2} . These are branched chain compounds.
 - c. Olefin series, C_nH_{2n} . These compounds make up only a very small fraction of most crudes. They are rather reactive and tend to polymerize and/or oxidize during storage. Important members are ethylene, propylene, butylene and higher members.
- 2) Ring compounds:
 - a. Naphthalene series, $C_n H_{2n}$. This series, which has the same formula as the olefins, differs in that its members are completely saturated compounds. Naphthalenes predominate in most gas oils and lubricating oils from all types of crudes and also in residual products. Examples are methylcyclopentane, cyclohexane, dimethylcyclopentane, and methylcyclohexane.
 - Aromatic (or benzene) series, C_nH_{2n-6} .

Cyclic hydrocarbons with elements other than carbon in the ring are called heterocyclic compounds, e.g., sulfur, nitrogen, or oxygen in the ring. Many hydrocarbons are complex, with their molecules consisting of groups of different hydrocarbon classes.

Crude oils are classified into three types on the basis of their general chemical composition:

- 1) Paraffin base. These crudes consist primarily of open chain compounds.
- 2) Naphthene base. These crudes consist primarily of cyclic (naphthene) compounds.
- Intermediate base. These crudes consist of large amounts of both

paraffin and naphthalene compounds.

Only small amounts of aromatic hydrocarbons are present in most crudes. However, some crudes do contain a significant fraction of aromatics, including benzene, toluene, and xylenes. Such crudes are referred to as aromatic base crudes.

Crude oils have long been divided into saleable cuts by fractionation in the refining operations. This is a separation by boiling ranges. ASTM standards contain the pertinent test methods and specifications for these products (ASTM, 1979). Table 2 lists the different types of petroleum products which are derived in this manner. Waxes and polar (oxygen-, sulfur-, and nitrogen-) hydrocarbons are largely removed during refining operations (Nelson, 1979).

The natural separation, into natural gas and ordinary crude, that takes place when petroleum leaves its underground reservoir is based on this principal. Natural gas is chiefly composed of paraffins from methane to pentane, CO2, No and sometimes He, but few, if any, unsaturated hydrocarbons.

Table 2. Different types of petroleum products

- A. Natural (casing-head) Gasoline Natural Gas
- в. Light Distillates
 - 1. Motor Gasoline
 - 2. Solvent Naphthas
 - Kerosene
 - 4. Light Heating Oils
- Intermediate Distillates
 - 1. Heavy Fuel Oils
 - 2. Diesel Fuel Oils
 - 3. Gas Oils
- D. Heavy Distillates

 - Heavy Mineral Oils
 Heavy Flotation Oils

 - Waxes
 Lubricating Oils*
- Residues
 - Lubricating Oils*
 Fuel Oils

 - 3. Petrolatum
 - 4. Asphalts
 - 5. Road Oils
 - 6. Petroleum Coke
- * Lubricating oil designations are based on viscosity and viscosity-temperature behavior.

range products. A light fraction consisting of C5-C6 hydrocarbons is produced, along with a naphtha fraction (boiling range 93-204°C). This includes naphthas, refined oils, aviation and motor gasoline, petroleum solvents, and jet fuel kerosene. Naphtha refers to any light oil product having properties intermediate between gas and kerosene.

Intermediate distillates are the medium boiling range products, with a boiling range of 204-343°C. These include: gas oil, heavy furnace oil (domestic), cracking stock, diesel fuel oil, adsorber oil, and distillates which are cracked and reformed to produce gas of adequate quality, i.e., the light C3 and C4 olefins. These distillates are often mixed with heavy tar to reduce its viscosity so that it may be marketed as fuel oil.

Fuel oils consist primarily of paraffins, iso-paraffins, aromatics, and naphthenes, plus related hydrocarbon derivatives of sulfur, nitrogen, and oxygen that were not removed by refining. Olefins are absent or negligible except where created by cracking or other severe refining. Vanadium and nickel compounds are low in volatility and do not distill into the No. 1 and No. 2 fuel oil fractions. Vacuum-tower distillates with a final boiling point equivalent to 454-566°C at atmospheric pressure are occasionally available as fuel oil not conforming with ASTM specifications and may contain 0.1-0.5 ppm vanadium and nickel in complex organic molecules, principally porphyrins.

Heavy distillates are the highest boiling range (343-566°C) product which is distilled off; they include: C20-C70 lubricating oils (which also come from residues), heavy oils for various purposes, and waxes. Lubricating oils contain very little olefinic unsaturation and some aromatic unsaturation. They are paraffins, cycloparaffins (multi-ring or condensed compounds), and aromatics (multi-ring or condensed compounds). Waxes are primarily straight and branched chain paraffins and 3-25% cycloparaffins. The remaining resins are very high molecular weight hydrocarbons (paraffins, cycloparaffins, and aromatics of various types and configurations). Lubricating oils are classified by the SAE system according to their viscosity and viscositytemperature characteristics. Oils can be classified according to their flash point, viscosity, pour point,

emulsibility, and resistance to sludging. Such information is extremely useful in ascertaining the usefulness of a particular oil.

Residues are the nondistillable pitch remaining after all the distillates are removed; they include: residual fuel oil, asphalt and petrolatum. Residues are very high molecular weight paraffins and ring compound hydrocarbons of various types and configurations, including some O, S, and N containing compounds in bridge or ring positions. They can be taken directly from the still and burned or thermally cracked (visbreaking) to reduce their viscosity. If the latter does not adequately reduce their viscosity, they may be mixed with intermediate distillates to achieve acceptable viscosity. Diluted with 5-20% distillate, this becomes No. 6 fuel oil, or it may be cut back with 20-50% distillate to make No. 4 and 5 fuel oils. However, distillates with inadequate solvent power will precipitate asphaltenes and other high molecular weight colloids from "visbroken" (severely heated) residuals.

No. 6 fuel oil contains 10-500 ppm vanadium and nickel in complex organic molecules, principally porphyrins, which cannot economically be refined out of the oil. Salt, sand, rust and dirt may also be present, giving No. 6 fuel oil a typical ash content of 0.01-0.5 weight percent. No. 4 and 5 fuel oils contain proportionately fewer contaminants.

The other types of refining operations involve chemical changes by conversion processes. Some of these processes are discussed below.

- Cracking or pyrolysis breaking down of large hydrocarbon molecules into smaller molecules by heat or catalytic action;
- 2) Polymerization linking of similar molecules, e.g., joining of light olefins;
- Alkylation union of an olefin with an aromatic or paraffin hydrocarbon;
- 4) Hydrogenation addition of a hydrogen;
- 5) Hydrocracking reduction in molecular weight;
- 6) Isomerization alteration of the atoms in a molecule without changing the number of atoms;
- 7) Reforming or aromatization rearrange molecular structure of hydrocarbons; particular objective is to upgrade naphthas to lower molecular weight molecules, i.e., motor fuels or aromatics.

Numerous other processes are used to derive additional hydrocarbons from petroleum fractions. The products and waste by-products from many of these processes can be used as flotation reagents.

Oil Characteristics

The molecular classification of an oil can be determined by clay-gel analysis or by carbon type analysis based on refractive analysis (Nelson, 1969). ASTM (1979 a,b) physical test data are frequently more readily available and the viscosity index or the viscosity-gravity constant (VGC) are indicative of the molecular characteristics of the oil, since each type has distinctive temperaturedensity-viscosity relationships. The viscosity index is calculated from the kinematic viscosities of unknown and reference oils at 40°C and 100°C. The viscosity-gravity constant is calculated from the viscosity at 37.8°C and the specific gravity at 15.5°C. This value is indeterminate for oils with kinematic viscosities below 3.5 cSt. Table 3 lists the characteristics of typical oil stocks.

The specific gravity of oils varies from 0.75 to 1.0, with the higher density fractions containing more high molecular weight hydrocarbons.

Similarly, the viscosity of the oils varies, being a function of the content of paraffins, naphthenes, aromatics, and their various derivatives. Paraffins are the least viscous of the hydrocarbons, even those with C20-C25 have a viscosity of 10-12 cs at 50°C. Thus, their addition to viscous oils decreases viscosity. Their viscosity usually increases with increased branching. The viscosity of cyclic compounds is greater than that of the paraffins and their effect on the viscosity of an oil is predominant. Viscosity increases as the number of rings in an aromatic or naphthenic compound increases; e.g., viscosity doubles as the number of rings goes from 1 to 2, and it increases by a factor of 6 as the number of rings goes from 2 to 3. The viscosity of a naphthenic compound is greater than that of an aromatic of the same structure; e.g., cyclohexane > cyclopentane > benzene. The use of various other chemicals can extend the range of usefulness of oils to lower temperatures and/or permit the use of higher molecular weight/viscosity oils.

CONDITIONING

The surface processes involved in preparing minerals for flotation vary widely in their dependence on agitation. Many of the reagents used are added to pump sumps, launders, flotation cells, or circuit feed boxes, with the turbulent flow conditions usually adequate without special conditioners. However, some reagent systems do require specific control of agitation intensities and reaction times to promote efficient reagent usage. Flotation systems employing either pure nonpolar oils or oil-surfactant systems to enhance surface hydrophobicity are in this category. For example, high energy conditioning has been reported to improve coal recovery and minimize reagent consumption in some cases. The agitation provides energy for oil breakdown into drops, suspension of particles and reagent drops in the pulp, and helps in the spreading of oil on the particle surface; and the retention time provides for adequate particle-oil drop interaction.

In some cases, over-conditioning can reduce process efficiency. Lower rank coals have a tendency to adsorb both nonionic frothers, such as MIBC, and more active frothers, such as cresylic acid, resulting in reduced concentration of frother in solution. In cases where frother concentration is significantly reduced due to such losses, increased conditioning can lead to reduced recovery. However, the addition of some frother with the nonpolar oil will improve drop formation and oil dropparticle interaction.

Conditioner geometry and agitation speed are important to the degree of efficiency with which they generate the necessary turbulence in the pulp and thereby control particle and oil drop suspension, oil drop formation, and the hydrodynamics of oil drop-particle interaction. The agitation energy is partly used by each of these processes.

In order to obtain a given homogenization of components in the pulp, a given quantity of energy is required in a particular case. Thus, for a given conditioner and retention time, the agitator will require a certain speed to provide the required degree of homogenization and adsorption; the greater the homogenization, the more rapid is the adsorption process. Particle degradation during conditioning is possible with some friable ores and must be minimized.

Table 3. Properties of typical base stocks (manufacturer's typical values) (Smit & Bhasin, 1985)

Chemical Properties	Amoco 11P	Texaco 1527	Calumet 200	Calumet 850	Calumet 1400	Calumet 6100	Calumet WF240	Cities 350	Cities 700 Cycle	Golden Bear 132 SAE 50	Golden Bear 2635-1 GB 142
Classification	-	-	naph.	naph.	naph.	naph.	naph.	para.	naph.	-	arom.
Viscosity at 37.8°C, cSt	21.6	9.7	44.4	187.2	304.6	10.5	2546	76.8	151.0	572	4844
Density at 15°C	0.88	33 0.904	0.904	0.912	0.915	0.890	0.943	0.879	0.918	0.96	0.974
Molecular weight	340	300	340	475	530	240	650	470	440	530	456
Viscosity-gravity constant	0.84	10 0.878	0.854	0.845	0.842	0.861	0.853	0.813	0.856	0.90	0.897
Viscosity index	70	62	49	58	63	41	38	95	50	-57	-103
Clay-gel analysis, weight %											
Asphaltenes	-	-	-	-	-	-	-	1.0	1.0	-	-
Polar Compounds	1.1	1.6	-	-	-	-	-	1.5	3.9	9.0	21.6
Aromatics	27.5	33.4	-	-	-	-	-	21.9	41.5	49.0	48.7
Saturates	71.4	65.0	-	-	-	-	-	76.6	54.6	42.0	29.7
Unsulfonated, wt%	82	83	-	-	-	-	-	-	_	-	-
Carbon type analyses, %*											
Ca	-	-	16	12	14	15	22	-	-	27	-
Cn	-	-	32	31	29	35	18	-	-	32	-
Ср	-	-	52	57	57	50	60	-	-	41	-
Sulfur, wt%	-	-	0.40	0.46	0.47	0.28	0.40	0.11	0.44	0.7	1.0
Pour point, °C	-32	-51	-34	-18	- 15	-51	+4	-12	-18	-1	+10
Flash point, °C	190	154	193	246	266	143	302	240	230	227	271

Table 3. (Cont.) Properties of typical base stocks (manufacturer's typical values) (Smit & Bhasin, 1985)

Properties	Golden Bear 2635-2 GB 148	Golden Bear 4053	Sunflex 107	Sunflex 115	Sunflex 160	Sunflex 2280	Sundex 750T	Sundex 7135T	Sundex 8600T
Classification	arom.	naph.	para.	para.	para.	para.	arom.	arom.	arom.
Viscosity at 37.8°C, cSt	2935	428.3	13.3	31.6	135.9	583	97.1	539	1855
Density at 15°C	0.960	0.919	0.847	0.864	0.876	0.893	0.959	0.964	0.963
Molecular weight	459	489	320	400	550	700	340	480	568
Viscosity-gravit constant	y 0.897	0.842	0.807	0.810	0.800	0.798	0.959	0.907	0.890
Viscosity index	-93	39	90	99	98	97	-8	-40	-
Clay-gel analysi weight %	s,								
Asphaltenes	-	-	-	-	-	-	-	-	-
Polar Compound	ls 17.9	3.0	0.2	0.4	1.0	3.8	5.2	12.0	15.9
Aromatics	43.5	28.0	7.9	12.0	17.1	21.5	66.5	62.3	57.9
Saturates	38.6	69.0	91.9	87.6	81.9	74.7	28.3	25.7	26.2
Unsulfonated, wt	68.0	-	-	-	-	-	-	-	-
Carbon type analyses, %*									
Ca	-	9	2	2	3	5	34	32	30
Cn	-	38	32	32	25	23	30	26	32
Ср	-	53	66	66	72	72	36	42	48
Sulfur, wt%	0.29	0.5	-	-	-	-	-	-	-
Pour point, °C	+2	-18	-20	-20	-15	-15	+2	+16	+35
Flash point, °C	274	249	182	207	266	302	204	243	302

^{*} Fraction of carbon atoms present in unsaturated, aromatic rings (Ca), in saturated, naphthenic rings (Cn), and in noncyclic, paraffinic chains (Cp).

Rate Controlling Processes

Because of the heterogeneity of the system it is clear that a large number of simultaneous and sequential steps must cooperate to bring about a final condition of optimal flotability. From the view point of both understanding and improving the operation, it is essential to identify those steps which are rate controlling. To accomplish this goal, each of the steps believed important is listed below and then discussed (after Arbiter and Williams, 1980):

- Reaction/equilibration of the nonpolar oil with pH and species in solution.
- Reaction/equilibration of particle surfaces with pH and species in solution.
- Decrease in size distribution of drops.
- 4) DLVO interaction followed by contact between oils drops and particle surfaces; with attachment dependent on surface characteristics of oil drops and particles.
- 5) Flocculation of particles in the micron size range and finer, due to Van der Waals interaction of hydrophobic surfaces and insufficient surface charge for electrostatic repulsion.
- 6) DLVO interaction followed by attachment of air bubbles to hydrophobic surfaces; bubbles being either entrained in the conditioned pulp or released from solution by turbulent pressure fluctuations.
- Spreading of oil lenses on individual particles after initial contact, and formation of multi-particle aggregates held by oil films and entrained air bubbles.
- 8) Application of shear stresses to initially formed aggregates resulting in reworking and exclusion of mechanically entrained gangue.
- Readjustment of surface surfactant levels to lower solution concentrations with greater selectivity developing.
- 10) Abrasion of surface collector films from particles where collector was adsorbed due to initially high levels and which may not respond to process 9 because of hysteresis.

The probable agitation energy requirements for these processes fall

into two ranges:

- Low energy processes, less than 0.2 HP/ft³; and
- 2) High energy processes, 0.2-0.5 HP/ft³.

The processes falling into the first group are discussed below, followed by a discussion of the second, with particular reference to their roles in developing optimum flotability.

Low Energy Processes: This category includes processes 1, 2, 4, 5, 6 and 9.

Processes 1, 2 and 9 are simple equilibrations of mineral surfaces with solutes. All of the data from laboratory studies and industrial operations indicates that only minimal agitation requirements are adequate for their completion. For example, complete mixing of a soluble reagent in a flotation cell requires seconds. Thus, for convective mixing of reagents and diffusion to surfaces through stationary layers to form monolayers or less, it is improbable that the power intensities under consideration are necessary. If there is hysteresis involved in collector adsorption, then process 9 may fall into a higher energy category.

Process 4, the contacting of oil drops and particle surfaces, requires very little conditioning provided that: the drop size distribution averages in the lower micron size range, as obtained by pre-mixing, and that only a single mineral is involved. If the drops are coarser, which will be the case if the oil is added to the conditioner without pre-mixing, the interaction will be less than optimum, because the coarser drops will be fewer in number and only a part of the mineral will be floated initially. Conditioning with too coarse a drop size may effect some improvement, but the results may not equal those with an appropriate initial drop size. This is significant in considering high agitation energy requirements, and is discussed further below.

Process 5, the simple flocculation of fine particles with hydrophobic surfaces, but without oil, requires little energy.

Process 6, the attachment of air bubbles to hydrophobic surfaces, requires lower agitation energy; flotation cells in larger sizes have power intensities from 0.05-0.10 HP/ft³. Furthermore, flotation is obtainable in very low intensity devices, such as the Hallimond tube or by gas precipitation in vacuum without agitation; or in some mineral systems with captive bubble apparatus.

High Energy Processes: This category includes processes 3, 7, 8, and 10.

Process 3, achieving the desired size distribution of nonpolar oils. Evidence from numerous investigations (e.g., Arbiter and Williams, 1980), suggests that the proper drop size should be achieved before, rather than during, the conditioning step for two reasons:

- 1) It is more effective to disperse nonpolar oils, with or without alkali, in a concentrated stock solution and with small equipment designed for the purpose;
- 2) As mentioned earlier, the recovery obtained with an initially coarse drop size is consistently poorer, and cannot be brought to the same level as that obtained with any reasonable amount of preconditioning.

 However, consideration must also be given to the average particle size of the mineral in this case; the finer the particle size of the feed, the greater the importance of predispersion of the nonpolar oil (Arbiter and williams, 1980).

Processes 7 and 8, the spreading of oil lenses and formation of multiparticle aggregates, followed by reworking and exclusion of gangue particles, respectively, are judged to be the processes most responsible for the high energy conditioning time requirement. The nonpolar oil, after optimum conditioning, produces not only a hydrophobic particle, ready for attachment to air bubbles in the flotation cell, but rather a multiparticle agglomerate including air bubbles.

Data from several investigations (e.g., Parkins and Shergold, 1976; Bensley et al., 1977; Seitz, 1979, 1986; Arbiter and Williams, 1980), clearly indicates that a major part of the conditioning process is associated with agglomerate formation, which is a dynamic process involving growth by collisions among free particles and nuclei, and then decomposition of agglomerates and their reformation by shear forces in the turbulent system. Meanwhile, the nonpolar oil concentration is changing, through interaction with particles, and surface concentrations are reflecting these changes. Depending on the speed of these changes, and on ore grade,

particle size, and gangue type, selectivity may be adequate at the peak of agglomeration or it may require further reworking of the agglomerates in order to reject sufficient gangue from the agglomerates.

Process 10, abrasion of surface reagent, may also be involved in order to increase selectivity. Evidence suggesting this possibility lies in the power intensities used in some conditioning studies and plants. The upper range is 0.5-0.7 HP/ft³, which is about the lower range for true attrition scrubbing.

PROCESS CONSIDERATIONS

Potential Benefits

A number of different benefits have been observed to result from the use of nonpolar oils as flotation reagents. All or only some of these benefits may be observed in any particular case. These benefits include the following:

- Reduction of collector dosage requirement. This results from co-adsorption of the oil and collector on mineral surfaces. As the nonpolar oils of interest are less expensive reagents (¢/lb.), a considerable cost saving can arise.
- 2) Increased rate of hydrophobic mineral recovery due to improved air bubble-particle adhesion. This can result in a more highly mineralized froth which drains more easily. It also reduces the initial entrainment of fine hydrophilic gangue by reducing the necessary retention time. These effects result in improved process selectivity.
- 3) In some cases, overfrothing due to the presence of slimes can be reduced or eliminated. This results from such phenomena as agglomeration of the slimes or lower collector dosages.
- 4) Coarse particle recovery can be improved without the attendant increase in recovery of undesirable particles which typically accompanies increased collector dosages. This effect is illustrated by the data shown in Figure 1, where increased fuel oil additions significantly increased the recovery of hard to float coarse coal particles without significantly increasing the recovery of gangue particles.

Implementation Program

The initial assessment of using nonpolar oils in any particular flotation circuit should consist of tests using No. 2 or No. 4 fuel oil to supplement the collector addition. In a series of tests, variable amounts of collector and fuel oil should be used and the grade-recovery response observed. Note that the use of a nonpolar oil will necessitate use of high energy conditioning or oil pre-emulsification in order to obtain the best performance. After conditions have been identified where nearly equivalent grade-recovery results are obtained, the use of alternative oils can be investigated. Selection of promising alternative oils can be based on mineral and oil surface behavior in solution and the nature of oil dropparticle interactions (e.g., see Smit and Bhasin, 1985; Seitz, 1986).

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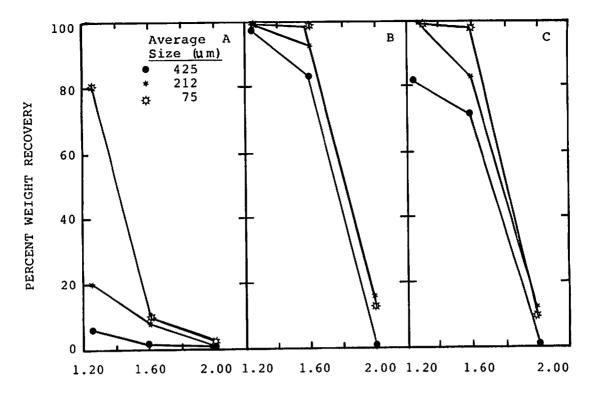
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AVERAGE PARTICLE SPECIFIC GRAVITY

Figure 1 . The Effect of Reagent Regime, Particle Size, and
Particle Composition (Specific Gravity) on Recovery
for a Lower Kittanning Seam Coal. Frother: 1 drop
MIBC. Collector: No. 2 Fuel Oil - A. 1 drop, B. 8
drops, and C.16 drops (Seitz, 1986).