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**DEVELOPMENT OF COMBINED
DRESSING-HYDROMETALLURGICAL PROCESSES**

by

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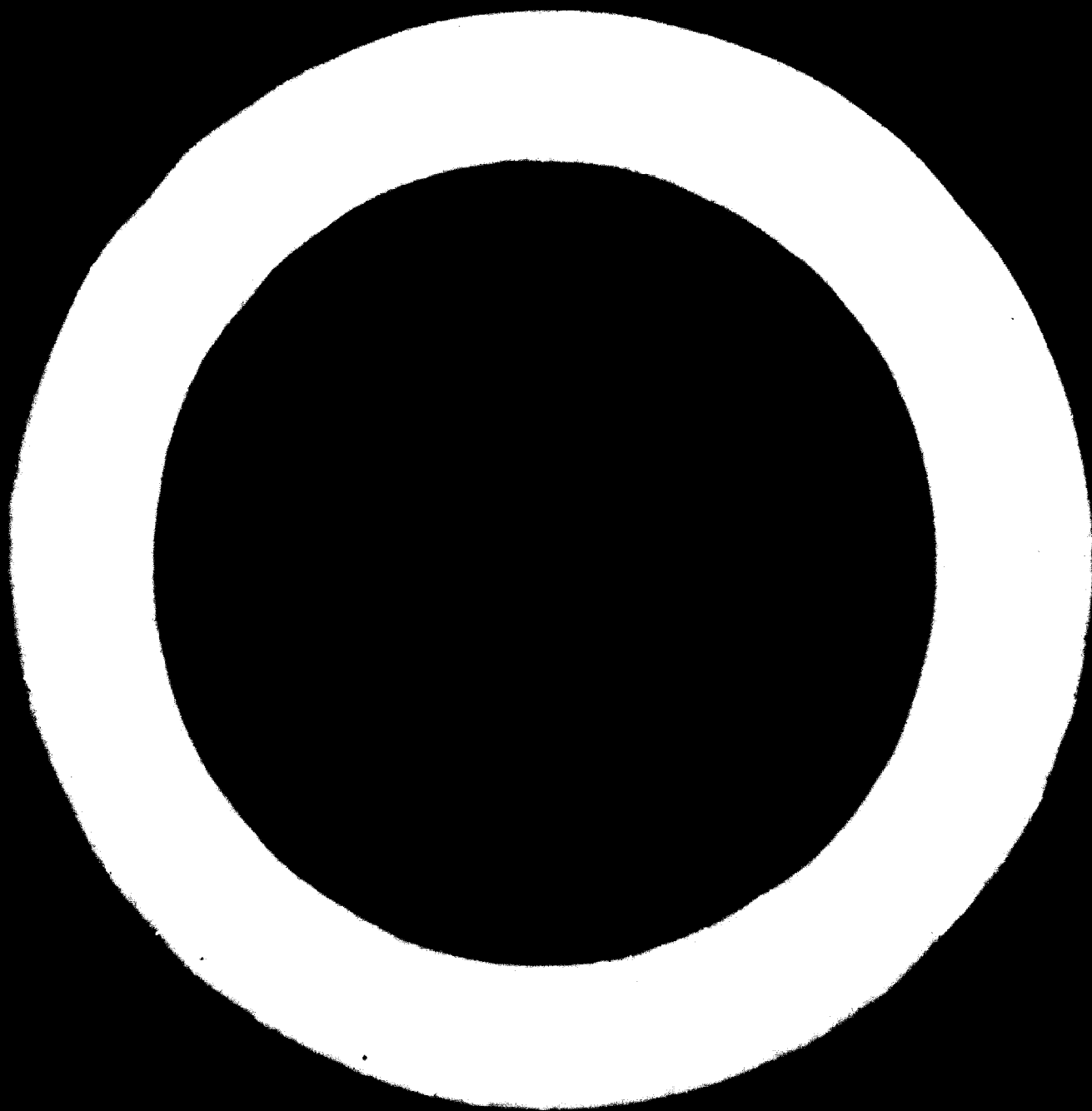
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Theses

The necessity to use combined dressing-hydrometallurgical processes is caused by the existence of such ore types from which it is technically impossible or economically unprofitable to extract copper with dressing or hydrometallurgical methods only. Oxidized or mixed (sulphide-oxidized) copper ores belong to such types.

A combined process usually consists of the following operations: 1) The conversion of oxidized copper compounds into a solution (leaching); 2) The extraction of copper from the solution; 3) Flootation of copper compounds insoluble in acid - sulphides and others. The succession of these operations may be different, that is a hydrometallurgical operation may precede floatation and vice versa.

Depending upon the ore mineralogical composition and physical characteristic, all the ore or only some part of it - sands or slimes or floatation products, may be subjected to all these operations.

Copper Leaching

Various solvents can be used for leaching oxidized copper compounds, sulphuric acid being the cheapest and most convenient

solvent.

The cost of sulphuric acid depends upon the technique to obtain it and it varies within 10-20 dollars per a ton. In practice less than 30% is used for copper solution, the rest of the acid being spent for the solution of the rock - carbonates, aluminium silicates iron compounds and others. Therefore, ores with high content of alkali-earth metal carbonates requiring 10 kg/t of sulphuric acid for each per cent of CaCO_3 or MgCO_3 , can not be treated by means of the processes including leaching with sulphuric acid.

If there is the so-called "refractory copper" in the ore it is necessary to use more concentrated solutions and heat up the pulp (Almalik, USSR); as a result acid consumption for solving barren rock and the residual acidity increase. On the average, acid consumption for non-carbonate ores is 4-5 kg for 1 kg of copper, and it rises up to 12 kg for 1 kg of copper for the ores with the increased content of carbonates (Djeskasgan, USSR).

The rate of leaching, and hence the apparatus volume depends upon the particle size and the leaching method.

Leaching method	Particle size, mm	Leaching duration
in vats	- 12	4.5-5 days
In drums with washing-off in classifiers	- 6	10-15 min
Agitation (slimes)	- 0.5	0.5-1 hour

Copper Extraction from Sulphuric-Acid Solutions

Electrolysis is applicable to rich solutions with copper content not lower than 20 gr/l. It is possible to obtain concentrated solutions when leaching products with high content of copper (concentrates, intermediate materials and others) or when processing lean ores by means of countercurrent percolation or decantation methods.

From lean solutions copper is extracted by cementation with iron scrap. Cemented concentrate with about 75% copper content is melted.

Copper separation from a solution directly by electrolysis is a more effective operation than cementation, melting and electrolysis both in capital and maintenance costs, but a considerable cost and the complexity of solution separation by countercurrent decantation prevent using a scheme with obtaining rich solutions applicable for electrolysis.

An extraction method of copper concentration from lean solutions with the successive separation of copper by electrolysis, is being developed.

LFP Process

The main process which makes possible to extract copper from a pulp is that of LFP: leached copper is cemented and floated.

Leaching is carried out in a series of vats (usually three) at pH of about 1.5 at the beginning and 2.3-2.5 in the end.

Cementation is effected either with scrap or with sponge iron dust, or with dust obtained by grinding iron chip in ball mills; the operation is carried out in a series of vats - cementaters. The least precipitator consumption is at $\text{pH} = 3$. Therefore in case of an increased acidity in the cementation vat supply the acid excess is neutralized with lime. The removal of the inert return scrap is done on screens and magnetic separators. Iron consumption for cementation is on the average 1.5 kg for 1 kg of copper. Scrap cost is 60 dollars a ton, but in a country having an undeveloped metallurgical industry scrap is less expensive - about 20 dollars a ton.

Cement copper floatation is mainly done using collectors not reacting with copper cation in the solution: dixanthogenides, minerecs, hydrolyzed aerofloats, Z-200 and others and more seldom with normal aerofloats and xanthater.

To prevent the solution of cement copper fine grinded iron must circulate in the pulp. Floatation reagents are added in the end of cementation, before aeration. Flotation of sulphides and cement copper is carried out separately or jointly.

LPF process is most effective as an additional one for extracting oxidized copper from mixed ores. The process is profitable if the amount of additionally extracted copper is 1 kg/t of ore.

Further increase in LPF process profitableness is possible by means of decreasing the cost of materials - acid and precipitator, improving the equipment and increasing its corrosion resistance (new alloys, plastics, rubber and others), decreasing the reagent costs, automation of control.

Attempts are being made to replace expensive iron with less expensive calcium sulphide and iron sulphide obtained by heating up pyrite with lime, precipitation with copper concentrate in autoclaves and others.

Sorption process

Of interest is the process of extracting copper from solution in the pulp by ion exchanger having sufficient capacity copper cation, sorption selectivity and mechanical strength.

Copper saturated ion-exchanger of 0.6 mm and larger in size are separated from ore particles of - 0.1 mm in size on 0.6 mm grids subject to countercurrent desorption with acid and the solutions with copper concentration of more than 20 gr/l are directed to electrolysis.

If the resin particles are fine then after saturation the resin can be filtered with ionogenic collector and also directed for copper desorption.

The regenerated resin is a return one.

This process disadvantages are a larger volume of the apparatus in leaching stage - sorption and the consumption of an expensive ion exchange resin; true, its consumption is supposed to be not more than 0.1 kg/t. The advantage is replacing pyrometallurgical processing with electrolysis which decreases considerably processing cost and saves acid consumption at the expense of the return electrolyte.

In case of inexpensive hydrogen copper can be precipitated

in the form of a high - quality powder in an autoclave.

With "floatation-sorption" combination oxidized copper extraction can be increased by more than 5% compared with the LPF process.

Other solvents instead of sulphuric acid are not yet of practical importance.

Ammonia process requires greater capital costs and hermetic apparatus; the application of this process does not increase. Leaching with alkali from carbonate ores is not effective enough.

The way to solve oxidised copper and sulphides - chalcocite and covellite - with cyanide but cyan process was used only for treating gold ores or gold containing tails of floatation mills with copper or pyrite concentrates extraction at the same time (Flin-Flon Mill).

Recently a method was suggested to extract copper from the sand of floatation tails by percolation in an original apparatus with the subsequent copper precipitation in the form of cuprus sulphide and cyanide regeneration. This method, however, is still in the stage of experimental tests; it has complex apparatus and technology and requires increased capital costs.

At present LPF process is the principal combined floatation-hydrometallurgical process to treat oxidized and mixed ores, and from year to year it is more extensively introduced into world practice.

The use of combined dressing-metallurgical processes is necessitated by the availability of such ore types from which it is technically impossible and economically unprofitable to extract copper only by dressing or hydrometallurgical processes. Oxidized or mixed (sulphide oxidized) copper ores belong to such types.

The basic method of dressing copper ores is flotation, but it does not make possible to extract copper in the forms of chrysocolla, cuprite or sorption copper bound with clays or iron hydroxides, etc. On the other hand, oxidized copper compounds dissolve well, for example, in sulphuric acid solutions, but the solution of well floating copper sulphides is a relatively complex operation and requires the selection of special solvents.

In case of great variety of copper ores as to their mineralogical composition and physical characteristics the schemes of combined processes of treating them, are various, as well.

The combined process usually consists of the following operations:

1. Transferring of oxidized copper compounds into a solution (leaching);
2. copper extraction from the solution;
3. flotation of insoluble in acid copper compounds--sulphides and others.

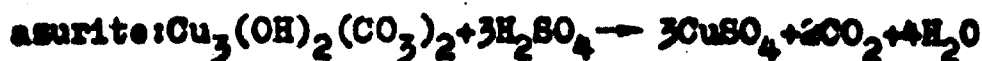
The succession of these operations may differ, i.e., a hydrometallurgical operation may precede flotation and vice versa.

Depending upon the ore mineralogical composition and physical characteristics all the ore or only some part of it may be subjected to all these operations.

Copper leaching

Different solvents can be used for leaching oxidized copper compounds, but sulphuric acid is the cheapest and the most convenient one.

The basic oxidized copper compounds in ores dissolve according to the following reactions:



and so on.

Sulphuric acid cost depends upon the method it is obtained by and varies within \$ 10-20 a ton. Less expensive sulphuric acid is obtained from SO_2 in exit gases of metallurgical works.

Theoretical weight rate is 1.5 units of acid per unit of copper. In practice less than 30% is used for dissolving of copper, the rest of the acid being consumed for rock solution-carbonates, aluminosilicates, iron compounds, etc. For instance, when leaching copper from the ores of Kalmaki deposit, USSR, /40/ acid consumption for leaching is in %:

copper	- 28.5
iron	- 28.5
Calcium and magnesium	- 12.4
aluminium	- 4.1
manganese	- 2.7
residual acidity	- 23.8

100.0

Therefore, ores with an increased content of alkali-earth metal carbonates requiring 10 kg/t of sulphuric acid for each per cent of CaCO_3 or MgCO_3 , can not be treated by the processes with sulphuric acid leaching. In practice the permissible consumption rate of acids is 3-5 kg for 1 kg of extracted copper. Usually at the beginning of copper leaching the solution pH = 1.5 and in the end it is 2-2.5. But in case of the "so-called" bound copper or copper which is difficult to transfer into solution the residual acidity must be increased up to 4 gr/l which involves additional acid consumption to decompose aluminosilicates and iron compounds.

The apparatus used in the leaching process is different depending upon the successive operation of copper extraction from the solution.

With purely hydrometallurgical processing of oxidized copper ores under favourable climatic conditions pile leaching is most widely spread. On filtering solutions through an ore bed one obtains relatively pure solutions from which rich concentrates of cement copper are obtained by iron cementation and intended for melting.

Pile leaching with the successive cementation is described in detail in the informative circular 8341 U.S. Bureau of Mines /1/.

In connection with the fact that economically it is more profitable to obtain cathodic copper as a commercial product, it is desirable to get more rich pure solutions applicable for electrolysis by means of leaching. When processing lean ores it is possible only with vat leaching by the percolation of the deslimed granular material or by means of counter-current decantation (CCD) on treating slimes. Both processes have found application.

Thus, for instance at Inspiration Consolidated Copper Co factory milled to 25 mm ore comes to screening. The under size product of 6 mm in size is passed into classifiers. The discharge of classifiers goes to hydrocyclones whose oversize product contains 85% - 74 μ and the sands are returned to the classifier. The oversize product of screening is re-

grinded on rolls up to the size of - 10 mm in a closed cycle with screens. This product is combined with sands from classifiers, it contains 85% - 74 and 7-8% of moisture. It is charged into 13 tanks (52.5x20.5x5 m), approximately 9,600 t into each of them. Leaching is carried out with the waste electrolyte heated up to 55°C and supplied at the rate of approximately 11 m³/sec. On the average the duration of leaching is 4.5 days.

After washing with water the ore containing undissolved copper sulphides is passed to a flotation factory.

Thus, oxidized copper is first extracted from a deslimed ore by leaching, then sulphide copper is extracted by flotation with a usual method. Desliming is necessary, for slimes sharply decrease the speed of the solution percolation and increase the duration of leaching process and ore washing after leaching. At the mill in Weed Heights (Anaconda Company) harmful effect of slimes on percolation is eliminated by water sprinkling the ore up to 5-7% moisture content in it on charging it into vats. In this case slimes adhere to big grains, form granules and distribute uniformly in the ore layers in the vats.

Slimes are treated in a reverse order: first sulphides are floated, then flotation tails are thickened up to 40% of solid content and the oxidized copper is leached with sulphuric acid in wooden agitators: 6 m in diameter equipped with mechanical mixers and air lifts for discharging the pulp. Slime leaching

duration ranges from 10 min to 1.5 hr. Separation of copper containing solution is effected by means of a countercurrent in 4 thickeners. 45 m in diameter.

The use of return electrolyte decreases acid consumption.

Acid consumption can be decreased if flotation of sulphides is first carried out with extraction easily floated oxidized copper minerals, such as malachite and azurite, and flotation tails are transferred for leaching. Thus, on dressing Almatik (USSR) oxidized ores the preliminary separation of sulphides and partially carbonates by flotation makes possible to decrease acid consumption by 25-30%. /41/.

Besides there is no necessity of using lime to neutralize the pulp before flotation.

Most often all the ore is leached. In some cases, however, when oxidized copper content is low the latter mainly transfers into slimes out of which copper is badly extracted by flotation. Therefore, only slimes are sent to combined processes. Sands are dressed by flotation (nutte mill). The presence of carbonates in the ore makes possible sharp decrease of acid consumption. Thus, in Djeskagan (USSR) using such a scheme decreased acid consumption two times.

Acid consumption can also be decreased if not the ore is leached but flotation products relatively poor in copper content. Thus, at Nchanga mill (Zambia) after separation of rich sulphide concentrates by flotation, oxidized copper and part of sulphides are extracted from tails by flotation into concentrate with 11.4% of oxidized copper and 1.7% sulphide copper

contents. Oxidized copper is dissolved with sulphuric acid out of this concentrate and the clarified solution passed to electrolysis.

After leaching cakes are fined and sulphides are floated.

When floating the oxidized copper compounds, alkali-metal carbonates remain in tails what sharply decreases acid consumption per unit copper.

Separating of solution from solid and obtaining of relatively pure copper solutions by means of countercurrent decantation in case of dressing slimes and pulverized products is a relatively expensive operation. Substantial investments are necessary to organize thickening. Therefore especially attractive are methods by which the dissolved copper is extracted directly from the pulp without separating liquid from solid.

In 1929-1930 Prof. Mostovich V.Ja. (USSR) /38,39/ developed a process on a laboratory scale consisting of the following operations: leaching - cementation - flotation. Simultaneously, during 1929-1934 F.W.Maclennan and Marmon Keyes developed independently an analogous LFF process and introduced it at Miami Copper Co. Mill (USA).

The process differs with the way of copper precipitation or cementation in solution- in pulp by means of metal iron according to the reaction:



cement copper is then floated.

The process has some technological advantages, especially

in cases when there are sulphides in the ore and pulverization is required to decompose their grains.

Flotation of sulphides can be carried out up to leaching operation and the tails after flotation are subjected to LFF process. Such scheme is realized, for instance, at Miami and Bagdad Mills (USA) and is also planned for a new section in Almalik (USSR).

At Miami Mill owing to the presence of molybdenum in the ore, copper and molybdenum sulphides are first floated from the ore containing 0.4% of sulphide copper and 0.5% of oxidized copper. Bulk copper - molybdenum concentrates are then divided into copper and molybdenum ones. The presence of cement, readily oxidized copper would make selective flotation more difficult. Therefore, oxidized copper is leached from sulphide flotation tails and cement copper and sulphide remains are floated.

In this case lime is added to the cement flotation tails to precipitate iron and copper salts up to pH 10.8 and reagents (pine oil, xanthate and alcohol B-23) and after three minutes' stirring sulphides are floated.

As mentioned above, a more finely grind ore is leached much faster (hundreds of times) than the ore in case of a pile percolation process of 100-150 mm in size, the apparatus volume decreasing correspondingly. Therefore, on preparing the ore for LFF process operation of leaching and separating the solution from the granular part can be simplified.

Thus, at Hyden Mill deslimed in hydrocyclons product of grinding in rod mills of - 6 mm in size, is leached with pH 1.5 in drums (3.6x7.6 m) during 10 min and washed by means of a counterflow in two acid-proof classifiers. Washed sands with lime adding are fined and floated to extract sulphide copper and discharge from classifiers are combined with slime fraction which is leached in mechanical wooden mixers 12 m in diameter, the duration of leaching slimes being 1 hour.

Acid consumption in this case is 3-4 kg/t of ore or 4 kg/kg of copper. 2/3 of total acid consumption are supplied to the drum for leaching sands, and 1/3 is supplied to the vat for leaching slimes.

At Xero Mill (Cyprus) leaching and countercurrent washing of material of - 13 mm in size are carried out in 4 classifiers.

The fine ore or slime are usually leached in three vats. This makes possible to keep the sulphuric acid concentration more accurately and regulate the process by the residual acidity, as well. The basic part of copper is dissolved in the first vat. At Almalik Mill (USSR) 95% of soluble copper transferred to the solution in the first vat during 30 min. leaching. In two other vats only about 2% additionally dissolved during an hour.

Copper Concentration

In case of hydrometallurgical treatment of oxidised lean ores, rich pure solutions with copper content not less than 20 gr/l are used for electrolysis, utilising waste electrolyte for leaching.

From lean solutions copper is extracted by a less economical method - cementation followed by melting of cement copper concentrates. Cementation is carried out in chutes /1/ with lattice bottom to remove cement copper. For the last time cementation is effected in cones designed by the firm Kennecott Copper Corp.

In LPF process cementation is effected in drums or in the pulp with stirring in vats-

At Rosita Mill (Nicaragua) cementation is carried out in drums (3.6x7.5 m) and at Miami Mill - in two drums (2.7x8.4) of wood boards. The drums rotate at a speed of 8 r.p.m.

Big iron scrap, mainly cans and also wastes of steel foundries and machine-building plants, etc. are used for cementation in chutes and drums.

To separate non-reacting scrap remains the pulp from drum cementators is supplied to stainless steel screens and magnetic separators. From screens and magnetic separators the scrap is returned into the process again.

With big scrap a large scrap weight has to be changed to produce sufficient reaction surface.

Scrap price depends upon that of steel and reaches 50 doll. a ton. Can scrap price is 20% higher /2/.

Can scrap crushed into plates in hammer crushers was used by Ohio Mill (USA).

Here scrap was periodically manually charged into cementators situated in stages to provide for the pulp gravity flow. From the last vat the pulp comes to a drum screen and then into

a magnetic separator for trapping the return scrap.

Fine scrap providing rapid copper cementation during 5-10 min. is required for cementation in vats.

Sponge iron obtained by reduction of iron ore or pyrite cinders and others, is a more active precipitator.

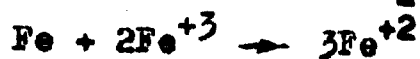
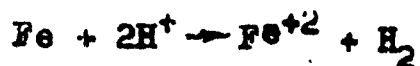
Thus, at Kennecott Copper Corp. Mills the necessary sulphuric acid is produced from the pyrite obtained from the process tails at Hyden Mill, and sponge iron is produced from pyrite cinder after its additional desulphurisation in Bruckner's furnaces using natural gas and soft coal as a reducing agent. The process of obtaining sponge iron from slag is also developed. The sponge iron is crushed in a closed cycle with a screen up to 35 mesh. Metal iron content is 50-60%. For Djezkazgan and Alma-lik Mills (USSR) sponge iron having activity up to 80% was produced from iron hematite ore in tube furnaces.

The cost of sponge iron for metal iron in the USA is about \$ 50 /3/.

Under conditions existing in the USSR iron chip crushed up to - 1 mm in a ball Mill, is a less expensive precipitator. At Karsakpaisk Mill (USSR) iron chip was crushed in a ball Mill in an open cycle /42/. The Mill product came into a classifier. The slimes of iron oxides and earth admixtures, oil, etc. were removed into dump together with the classifier discharge, coarser iron which was passed into the beginning of cementation was separated from the classifier top part, and finer iron was separated from the classified bottom part before the discharge threshold and it was transferred into the end of cementation for copper complete settling.

Cementation is usually effected in vats connected in series. At Altin-Topkhansk Mill (USSR) 78% of copper were settled from solution in the first vat for 6 min., 90% of copper was settled in 2 vats and 99% - in 3 vats. At Djezkazgan and Almalik Mills they went over to cementation in flotation machine chambers 6 m³ in volume with decreased ineller speed and without air section.

Theoretical iron consumption for copper cementation is 7/8 of copper weight or about 0.9 kg for 1 kg of copper. In practice iron consumption is 1.5-2 times as much which is caused by secondary reactions:



Fine cement copper is readily oxidized by air oxygen:



and iron must again be spent for cementation.

All the reactions given above depend on the pulp pH.

Optimal acidity for cementation at which the least iron consumption is characterized by pH = 2.8-3. As the acidity in the last cementation vat is usually higher, acid excess is neutralized with lime. At Bagdad Mill a small conditioner is installed between the last leaching vat and cementation one to control pH.

At Miami Mill the last leaching vat discharge contains 2.3

gr/l of free sulphuric acid.

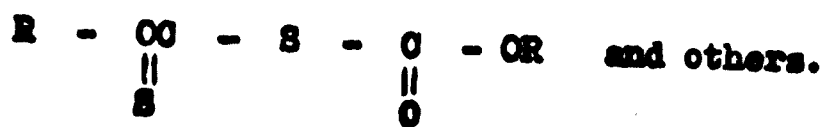
When mineral atacamite, $\text{CuCl}_2 \cdot 3\text{Cu}(\text{OH})_2$ is present in the ore ion Cl^- concentration in the solution increases. This accelerates the cementation process but at the same time improves copper and iron oxidation reactions, which requires an especially exact control in observing cementation conditions, such as: precipitator supply, cementation duration, pH of the medium, etc. and cement copper flotation conditions, as well /43/.

To maintain the reductive medium iron must be supplied twice: at the beginning and before the end of cementation, to prevent the increase of copper content in the solution. There must not be any aeration in the process and air lifts must not be used for mixing and transport. Best of all to transport pulp from one apparatus to another by gravity flow.

Cement Copper Flotation

Both ionogenic and non-ionogenic collectors are used for cement copper flotation. In American practice the following non-ionogenic collectors are used:

dixanthogenates, Minerecs A and B, which apparently represents mixed xanthogenate thioanhydrides:



Minerec composition at Miami Mill is 94 gr/t. At Rosita Mill in case of 36 gr/t consumption Minerec A is used with aerofloat 25 with 0.5kg/t consumption Carbomethoxy ester of butyl xanthogenic acid is an effective collector as well as Minerec A in

case of cement copper flotation. Cement copper floats well with reagent Z - 200.

In the USSR aerofloats with xanthate additions or without them with foamer addition, are used for cement copper flotation.

At Ohio Mill (USA) 45 gr/t of Minerec B are added into leaching, and only foamers are introduced into flotation.

At Tzukita Mill (Japan) ethyl xanthate - 85 gr/t and amyl xanthate - 55 gr/t are used.

Best collectors are those which do not form precipitates with blue vitriol. As Cu^{2+} ions are always present in the pulp in case of cement copper flotation, the consumption of reagents reacting with Cu^{2+} ions sharply increases.

Interacting with Cu^{2+} both xanthates and aerofloats form unstable salts which decompose into monovalent copper salts and disulphides that are good collectors of cement copper if they are present in the pulp in the form of fine emulsion.

The so-called hydrolyzed aerofloat - $(\text{RO})_2\text{P} \begin{matrix} \text{=O} \\ \text{SH} \end{matrix}$ or $(\text{RO})_2\text{P} \begin{matrix} \text{=S} \\ \text{OH} \end{matrix}$ does not form precipitates with Cu^{2+} and is, therefore, a more efficient collector than normal aerofloats whose efficiencies depend upon hydrolyzed aerofloat contents in them.

For better use of collectors minimum Cu^{2+} content in the pulp is necessary. Copper cementation is continued up to residual Cu^{2+} concentration of 0.02-0.04 gr/l.

With aeration during flotation oxidation reactions intensify and copper content in the solution can increase. To prevent

this metal iron with fresh surface must always circulate in the pulp. Besides, the collector must be supplied before the completion of cementation for the contact of copper particles with the collector to occur without air access.

At a number of mills small amounts of sponge iron are added into basic flotations to prevent copper oxidation.

Cement copper flotation schemes are simple: basic and control flotations with scavenging of rough concentrate. These schemes differ mainly in the way of finishing intermediate product - froth of control flotation and scavenging tails. There is an increased content of soluble copper and slimes in intermediate products and their return into basic flotation often causes violation or deterioration of cement copper flotation process. At Djezkazgan Mill the intermediate product is returned into the last leaching vat; at Big Butte Mill sponge iron is added to the scavenging and scavenging tails go into dump in spite of the increased copper content in them.

At Bagdad Mill final concentrate is taken at once during 2.5 min and the intermediate product is supplied to basic flotation. At Phaiso Mill they also first separate final concentrate, but the intermediate product is scavenged and the scavenging tails go into dump.

Flotation tails contain residual metal iron with a copper film. Therefore, as a rule, this iron is separated in magnetic separators and returned into the process. It is expedient to regrind this iron with sponge iron to renew metal surface and remove copper.

pH control is of great importance.

At most of the mills flotation is carried out at pH 4-4.5 and controlled by adding lime before flotation or by acid addition before the control flotation.

In some cases, however, even without lime addition the acidity sharply decreases in the process of cementation and especially in that of flotation. For example, when treating Djeskazgan ore (USSR) the pulp acidity decreases rapidly down to pH 5, especially in case of returning the intermediate product into the basic flotation. Therefore, to regulate pH sulphuric acid must additionally be supplied on the intermediate product must go to the last leaching vat.

The field of a combined process application depends upon a number of factors and first of all upon the ore nature and copper and noble metals in it.

Combined processes provide for an additional copper extraction and a more complex utilization of ores.

Economic efficiency of LPF process is higher in case of using it as an auxiliary process to the main flotation one.

According to practice adding a combined LPF process to flotation dressing is profitable if 1 kg of copper is extracted per 1 ton of ore (Hyden, Big Butte, Bagdad and others) and even 0.5 kg Cu/t ore (Morenci).

In Djeskazgan, Almalik (USSR) Mostovich's process for oxidised ores increases copper extraction by 20-50%. If the process is used as the only one the cost per ton of ore treating is much higher than that of flotation, and capital costs are also greater by 6-10%; hence profitable copper content

in the ore for LPP process must be approximately 1.5 times higher than for that of flotation only.

The disadvantage of a single hydrometallurgical process is losses of gold in tails.

According to N. Arbeter's /2/ data capital costs for the leaching - electrolysis process are approximately 6% higher, though copper ultimate production cost without taking gold into account, will be about 40% lower.

From American practice per ton of ore costs for LPP process in dollars are /3/:

H_2SO_4		(25 kg/t)
sponge iron from cinder	0.645	(8 50 t Fe)
flotation reagents	0.150	
amertisation	0.342	
Gastos Fundicion	0.500	

in all 1.764

Improvement and Development of New Combined Processes

Researchers and inventors tirelessly work at improving combined flotation-hydrometallurgical methods.

For ores dressed with difficulty, especially for extracting oxidized copper associated with sulphide copper and insoluble copper minerals of barren rock a method is suggested /5/ to treat crushed ore (10 mm in size) with concentrated sulphuric acid up to moisture content from 5 to 20% during ore transporta-

tion to storage bunkers with subsequent 6-8% addition of water. At pH = 1-2 the ore is treated during 1-3 hrs. after that calcium or iron sulphides or calcium sulphide and metal iron are introduced into the pulp to precipitate copper in the form of a sulphide.

The precipitator is obtained by heating pyrite and lime in a neutral or weakly reducing medium for 1-3 hrs at 815-870°C. The product cooled without air access contains 60% CaS, 25% Fe, 5% FeS + 7% CaO. +)

Precipitation lasts 2-3 min. (pH = 1.5) the precipitator consumption is 0.35-0.4 gr per 1 gr. of copper. Copper precipitated in the form of sulphide is floated together with sulphide primary copper at pH less than 7. This method makes it possible to decrease acid consumption and water consumption for leaching, to lower the amount of fine slime which is formed on dissolving the ore, to lessen the equipment volume.

At Morensi Mill precipitation by means of above precipitator was carried out simultaneously with leaching of 30 mm ground ore in the drums, installed behind the mill. The ore from drums mixed with precipitated sulphide copper and sulphidized surface cement copper are sent to the mills and after grinding is floated.

To improve the process of copper cementation on iron, and decrease iron consumption it is suggested /6,7/ to introduce special additives - inhibitors - organic compounds, containing nitrogen or sulphur, coco-nut oil, for example /6/. Inhibitors protect iron surface against sulphuric acid action but do not

*) At Morensi /4/ calcium sulphide is used for copper cementation instead of iron.

react with iron, eliminate iron oxidation and hydrolysis of oxide compounds. Inhibitor consumption is 0.5 kg per 1 t of solution.

To precipitate copper from solution it is recommended to use copper concentrate containing chalcopyrite /8/ by reaction



The process is carried out at the temperature of 160°C.

In the USA a method of copper extraction from oxidized carbonatite ores with sodium hydroxide is developed. The ore is crushed up to 10 mm size and leached in a drum rotating at the speed of 0.35 r.p.m. The pulp is filtered on a vacuum filter, the cake is washed with sodium hydroxide solution. Copper is precipitated from the solution by electrolysis at the temperature of 80°C, current density of 8 a/dm², bath voltage of 2-3 v.

The sponge containing 80% of copper settles on the cathode. The sponge is separated from the solution on a rotating vacuum filter, washed and remelted in crucible furnaces at 1250°-1260°C with small additions of a reducing agent. 99.5% copper obtained is poured into anodes and subjected to electrolysis. The waste electrolyte and evaporated washing water are returned for leaching and regenerated up to 95% of sodium hydroxide. The process was tested on a semi-industrial scale with oxidized ores containing 2.3-2.8% of copper and great quantities of alkali-earth metal carbonates, it provides for 88-92% extraction.

When treating mixed ores, cakes after leaching are regrinded and floated which makes possible to extract up to 90% of sulphide copper insoluble in sodium hydroxide.

A number of improvements in ammonia-carbonate leaching were suggested /11, 12, 13/ for extracting copper from sulphide minerals at the expense of introducing oxygen and catalysts: divalent copper ions, sulphite and thiosulphate on heating. The following process optimal parameters are recommended: NH_4OH concentration - 60-120 gr/l, $(\text{NH}_4)_2\text{CO}_3$ - 20-50 gr/l, Cu^{+2} - 10-50 gr/l, CuS (solid) - 10-50 gr/l, sulphite $(\text{SO}_3)^{-2}$ or thio-sulphite $(\text{S}_2\text{O}_3)^{-2}$ ions - 0.3-5.0 gr/l; rate of oxygen supply 2 1/2 of oxygen for 1 copper atom in a sulphide material; duration - 2 hours; temperature - 45-60°C. In this case copper extraction from chalcocite is 90%.

For the last years much work was done, with firms "Gaspé", "Noranda" and "Galiger" /8/ participating, to find a rational method of treating the ores of copper deposit Copper Maynten (Quebec, Canada) having 0.75% copper content. Basic useful mineral in oxidized ores is malachite, there is some copper in chrysocolla and chalcopyrite; in subordinate amounts it is contained in cuprite, tenorite and in a native copper form. Malachite associates to a considerable degree with limonite. Total amount of iron in oxidized ores is within 6%. Basic minerals of barren rock in oxidized ores are silicates, however these ores, on the average, contain 8% CaO and 4% MgO , as well.

The method using alkali leaching with sodium hydroxide did

not give positive results. Copper extraction was 50%.

Ammonia leaching method was rejected in view of great capital costs necessary to realize it.

LPF process with the application sulphide copper concentrate for settling copper did not provide for high extraction. Besides, there is no industrial apparatus for settling copper from pulps at high temperatures.

Design works were carried out in three versions:

1. Agitation leaching, filtration and settling copper from the solution with a sulphide concentrate;
2. percolation leaching, settling copper from the solution with a sulphide concentrate;
3. LPF process.

Agitation leaching with settling copper by sulphide concentrate (see Table 1) proved to be the most efficient for treating oxidized copper ores of Copper Maynten deposit.

With market price of 37 cents a pound the total amount of oxidized ores with 0.75% copper content can be treated by this method with economically profitable result.

For the last years cyanide methods for extracting copper from flotation tails, oxidized and mixed ores are greatly attracting researchers' interests.

A number of patents were published /14-15/ on the method of cyanide leaching copper from ore wastes, oxidized and mixed ores and on settling copper from solution in the form of cyanide with the subsequent treatment of the precipitate to obtain metal.

All of them involve copper transfer into a solution in the form of cyanide complex with the subsequent settling copper from semisulphide copper solution, and some regeneration of the reagent used to decrease the process cost.

In 1964, White Pine Copper firm built a pilot plant with the production of 12-24 t/day at its dressing mill in Upper Penon Sula (USA, State Michigan) to prove the possibility of copper extraction from copper flotation tails by means of cyanide method. The scheme of the apparatus line is given in Fig. 1.

Sand fraction of tails with average copper content of 0.24%, sizes 65% + 200 mesh, 10% + 325 mesh, 25% - 325 mesh, 40% moisture content, was treated.

Table 1

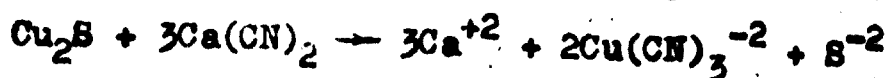
Technological and Technico-Economical Indices of Processing Oxidized Copper Ores of Deposit (Canada) by Different Schemes

	S c h e m e					
	L P F		Agitation leaching, copper settling from the filtrate with sulphide copper concentrate	Percolation leaching, copper settling from the solution with sulphide copper concentrate		
	1	2	3	4		
1. Copper content in the ore, %	0.75	1.0	0.75	1.0	0.68	0.91
2. Ore size for leaching	60% -200mesh		60%-200 mesh	15.9mm-50mesh		

	1	2	3	4		
3. Leaching conditions						
solid content	60		60			
temperature, °C	surroundings		surroundings	surroundings		
concentration, %	5		5	10		
H ₂ SO ₄ consumption, kg/t of ore	35		35	35		
4. Settling conditions						
temperature, °C	surroundings		160	160		
precipitator consumption, kg/kg						
copper						
Fe powder	2.0					
sulphide copper concentrate			30	30		
5. Cu extraction, %	75		75	65		
6. The amount of ore for treating, mill.t.	9	4.5	9	4.5	7.2	3.6
7. Total cost of recovery and remaking, dollars/t of ore	4.0	4.9	3.8	4.95	3.95	4.2
8. Total market cost of extracted copper with the price of 30 cents a pound	3.7	4.9	3.7	4.5	3.0	4.0
profit (+)	-0.3	0	-0.1	+0.35	+0.95	-0.2
loss (-)						
9. Total market cost of extracted copper with the price of 37 cents a pound	4.5	6.0	4.5	6.0	3.65	4.9
profit (+)						
loss (-)	0.5	1.1	0.7	1.45	0.1	0.7

Percolation leaching was effected in a vat (see Fig. 2) which represents a closed cylindrical container 2.44 m in diameter with a filter (4) located 15-25 cm above a cone bottom. Sands were supplied into the vat through a rotating distributor device /2/. After leaching the sands were discharged through the ring /3/ into the discharge pipe /6/ 30.5 cm in diameter through the vat centre up to the filter level. Along the vat periphery water nozzles are located to wash out sands. For solution seepage through the layer of solid on the filter the latter was connected to the vacuum line (10).

Leaching took place with pH = 11 by reaction:



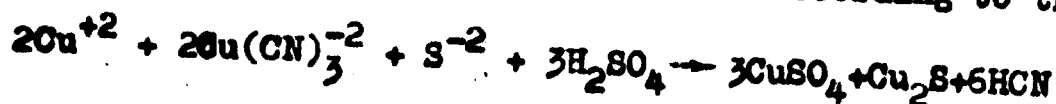
The process was controlled by the solution electric conductivity.

At the beginning of leaching the solution with low copper content came from vat /3/ into a special tank of diluted solution /7/. The necessary concentration having been reached, the flow was switched to the basic solution collector /6/. With the decrease in copper concentration and the increase in that of cyanion, sands becoming lean, the flow was switched to recirculation /5/. Waste sands were washed with water. When coming electrical conductivity decreased and this was the signal to switch the flow to diluted solution collector. When cyanid concentration in this solution became lower than it is permissible for economical regeneration, it was discharged. To decompose cyan-ion solutions must be treated with chlorine.

Leaching duration, charging of 15 t of tails and discharging includes, was 12 hours. Copper contents in solutions were, in gr/l: rich solution - 5.4; return solution - 1.5; lean solution - 0.7; cyan-ion counting for NaCl, gr/equiv./l: rich solution - 16.5; return solution - 17.2; lean solution - 3.2.

Two methods were tested for copper extracting from solutions:

1. Copper precipitation in the form of a sulphide by means of acidification with sulphuric acid according to the reaction:



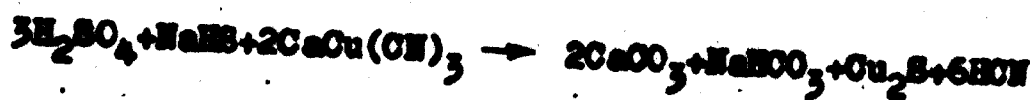
The process takes place in precipitator (10) at pH 2.3-3.0 with 10% sulphide excess of a theoretically required amount. To maintain the necessary sulphide concentration sodium bisulphate was introduced into the solution.

Practically complete copper settling was obtained.

The process occurring practically without any heating and all the apparatus being closed and connected to a ventilation system, poisoning with hydrogen cyanide was eliminated.

It was established that cake consisting of copper sulphide and calcium sulphate, can, equally well, be melted together with the concentrate /21/ or floated and the resulted concentrate can further be treated.

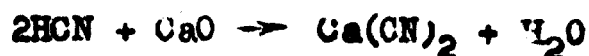
2. Copper separation by carbonisation of solutions by reaction:



When treating with carbon dioxide in carbonizer /9/ the

solution pH decreased up to 5.7, 60% of copper and 70% of calcium were settled. The pulp was separated in hydrocyclons, the liquid part after reinforcement was fed into the return for leaching.

This method disadvantage is settling calcium carbonate in pipelines, which makes a continuous process difficult. Off gases from reactors went to towers /13/ where nitrogen was supplied as a counterflow. Hydrogen cyanide was distilled off into absorber /14/ where it was absorbed with lime. The absorber contents was pumped into collector /8/ where return solution was fed and unslaked lime was supplied to regenerate basic reagent by equation:



Copper extraction into sulphide concentrate reached 91.6%.

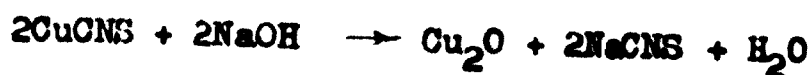
Reagent consumptions for extraction of 1 kg of copper were: lime - 2 kg, 75% H_2SO_4 - 3.6 kg, sodium bisulphate - 0.99. Cyanide regeneration was 43.7%, calcium cyanide activity was 48-50%.

Expressed in money the consumption of reagents was 10-20 cents for 1 kg of copper extracted, but capital costs are large, approximately 1.5-2 times higher than expenditures for a dressing mill. However, the method is being studied as for its application to Michigan's silicate ores /24/.

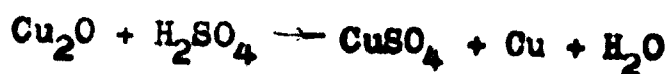
It is suggested to use organic products, for example, lactonitrile obtained as a by-product of acrylonitril, for cyanating /25/.

Dow Chemical Co. firm /23/ suggested a technique of copper extraction from a solution in the form of thiocyanate.

Monovalent copper thiocyanate is treated with alkali and basic reagent is regenerated:



In acid medium disproportion occurs with elementary copper formation:



The technique was tested for extracting copper from solutions after leaching mixed ores; extraction achieved was 92%; thiocyanate regeneration was 97%.

The firm studies different modifications of the method, thiocyanate flotation from the pulp among them.

On treating oxidized and mixed ores ion exchange processes for copper extraction from a solution without separating liquid from solid, are perspective trends.

A number of patents were offered in USA, England, France, Canada, Japan and other countries on sorption extraction of metals, that of copper from pulps, in particular /25, 26/.

Bagdad Copper firm is carrying on studies of copper extraction from its solutions using ion exchange resins.

A considerable complex of investigations was.

Many research works were carried out in the USSR on developing flotation - sorption - non-filtration method of extracting copper from oxidized and mixed ores.

According to this process a finely-divided ore (70-80% -

- 74%), intermediate product or lean concentrate are leached with sulphuric acid (or some other solvent) in contact with an ion-exchange resin (sorber).

In case of leaching in contact with a sorbent a more complete extraction of copper from an ore into solution takes place in the pulp, for as a result of copper sorption by resin the equilibrium between copper concentrations in solid and liquid phases changes. Great difference in particle sizes of the pulp solid (-0.1 mm) and of the sorbent (+0.5 mm) provides for their efficient separation after sorption on the grid or in the upward flow.

Instead of mechanical separation of a copper saturated sorbent it is possible to separate it by flotation adding a cation sorbent of amine type or active cation promoters with active aminogroups /35,37/ (Process LP/).

To effect the process of sorption from pulps in other branches of industry contact sorption apparatus with pneumatic stirring and grid drain are most widely spread.

Distinguishing features of treating a pulp in apparatus with a pneumatic stirring are the following: small ion-exchanger loading at a time, apparatus simplicity, a complete continuity of the process, the possibility to treat pulps with any density and viscosity /27/.

Fig. 3 gives a scheme of a pulp sorption treating in apparatus with pneumatic stirring, and in Fig. 4 a general view of a sorption apparatus with pneumatic stirring and a ring drain of

"inclined grid" is shown.

The pulp is supplied into the apparatus bottom part through a distribution drain at a speed providing for keeping the ion-exchanger in suspension. The resin is loaded into the top part. The ion-exchanger and pulp mixing is effected by means of the central airlift, another airlift being used to supply the pulp with ion-exchanger onto the separating grid of stainless steel or synthetic material. The pulp and the resin move as counter-currents. The type of the ore and metal concentration predetermines the number of apparatus installed. Resin desorption is also effected by a countercurrent scheme.

Ion exchange resin used in the process must be copper selective, have a sufficient capacity and mechanical strength providing for minimum ion-exchanger losses.

The aluate can be straight directed for electrolysis or for obtaining copper powder by hydrogen reduction which is also the process advantage.

In the USSR tests on flotation-sorption-electrolysis process of treating oxidized and mixed ores were carried out /28/.

The ore was crushed and ground in a closed cycle with classification up to 75-90% - 74μ and supplied for flotation of sulphides by a usual scheme, with copper concentrate being obtained after two recleanings. Flotation tails were leached with sulphuric acid at pH 1.5-3.5, solid to liquid content ratio 1:3, during 30-60 min., without heating. Depending on the grade of ores treated sulphuric acid consumption is from 30 to 60 kg/t of ore.

For copper sorption a modified an aminocarboxyl ion-exchanger having 98% mechanical strength. Sorption was effected in 8-10 stages, each of them lasting 20-30 min. The copper absorption resin capacity was 90-110 mg/gr. Copper saturated resin was separated from the pulp by draining on a grid with subsequent washing on a vibrating screen. Depending on the type of treated ores copper content in sorption leaching tails was from 0.07 to 0.18%; residual copper content in the liquid phase was of the order of 10-30 mg/l.

Copper desorption from the resin and ion-exchanger regeneration were carried out with 10-30% sulphuric acid in 3-4 stages, after which the resin was washed off from the acid in 5-6 regeneration columns with obtaining return solution. Residual copper content in the resin did not exceed 0.01%, copper desorption completeness being more than 99%.

The commercial regenerate contained up to 50 gr/l of copper, 100-150 gr/l of sulphuric acid and 2-6 gr/l of trivalent iron.

To reduce $Fe^{+3} \rightarrow Fe^{+2}$ it was treated with a reductant (SO_2) after which it was supplied for electrolytic separation of copper with insoluble anodes. The electrolysis was effected with a diaphragm in two stages, and commercial cathode copper was obtained. Power consumption for the electrolysis was 1700.-180 kwhr/t of copper.

In table 2 approximate comparative indices of oxidised copper ore treatments according to the two schemes are given:

1. leaching-cementation-floatation (LFF process)
2. floatation-sorption leaching-electrolysis.

Approximate comparison of the two schemes shows the sorption technology efficiency. The efficiency is due to the 8-17% increase in copper extraction (depending on ore types) the 10% and more decrease in production expenses per ton of copper, reduction of pyrometallurgical conversion volume at the expense of the decrease in the quantity of floatation copper concentrate treatment.

Table 2

Comparative Indices of Oxidized Copper Ore Treatments

Description	Combined process: leaching-cementation floatation		Combined process: floatation-sorption leaching-electrolysis	
	ore No 1	ore No 2	ore No 1	ore No 2
1	2	3	4	5
1. copper content in the initial ore, %	1.2	0.8	1.2	0.8
in it:				
sulphide, %	0.53	0.3	0.53	0.3
oxidized, %	0.63	0.5	0.63	0.5
2. Floatation copper concentrate output, %	4.0	3.0	3.0	2.8
3. Copper extraction into floatation copper concentrate, %	75 ^x)	70	52	40
4. Copper extraction into commercial regenerate, %	-	-	40	40
5. Copper extraction from the initial ore into cathode copper, %	73	68	90	73

	1	2	3	4	5
6. Consumption per ton of ore:					
sulphuric acid, kg	35.0	35.0	60	30	
sponge iron, kg	10.0	15.0	-	-	
orbent, kg	-	-	0.1	0.1	
7. Specific investment per ton of copper, %					
	100 ^{xx)}	100 ^{xx)}	90	104.5	
8. Production expenses per ton of copper, %					
	100 ^{xx)}	100 ^{xx)}	80	90	

Notes:

x) Only slimes (-74 μ) were used for LFF, sands were dressed by floatation.

xx) The common extraction is given .
Capital and maintenance costs are given taking into account the construction of metallurgical conversion for treating float-concentrates.

The process economical efficiency can be increased by obtaining high purity copper powder from commercial regenerates.

The results obtained served the basis for a design adaptation of floatation- - sorption - electrolytic process for the USSR oxidized and mixed ores.

A number of the USA firms are carrying on the work on replacing a cementation method of copper extraction from solutions by that of extraction with the subsequent copper precipitation from the reextract by electrolysis or by hydrogen reduction under pressure.

Americal firm General Mills /29-31/ developed a copper selective extracting agent hix - 64 whose price is 5.5 dollars for 1 kg, acting in acid mediums (pH = 1.5-3).

This extragent disadvantage is its small copper absorption capacity (2.5 gr/l in a 10% solution).

From 1965-66 pilot plants at Inspiration /32/, Esperanza (Duval firm) /33/, Bagdad Mills work using a 6-10% hix-64 solution in kerosene. In 1968 a copper selective extragent - Sx was tested at the pilot plant of Blue Bird Mine Mill /33, 34/.

Up to the present time, however, there are no sufficient data to determine the industrial usefulness of extraction copper separation.

Conclusion

Ever increasing demand for copper causes to decrease marginal copper content in ores, involve into treatment ores with high content of oxidized minerals. In this connection combined

processes using hydrometallurgical methods acquire still greater significance for treating copper raw materials. LPF is the basic combined process as an auxiliary one for additional extraction of oxidized copper from mixed lean ores having been dressed by floatation. In a number of cases its application to non-carbonate ores is profitable, if additional copper extraction is not less than 1 kg for 1 t of the ore.

The process can be used for finishing lean copper concentrate and intermediate products containing oxidized copper.

The increase in economic efficiency of this process is associated with the decrease in acid, iron, and floatation reagents costs, the increase in the equipment anticorrosion stability and introduction of the process automatic control.

A possible LPF competitor is a combined process: floatation-sorption with ionite from pulps obtaining solutions applicable for electrolysis or getting high-grade copper powder by hydrogen reduction.

This process is, however, still in the stage of experimental tests, requires increased capital costs and the availability of relatively cheap and mechanically strong ionite - copper sorbent.

Extraction processes of obtaining copper from lean solutions which make it possible to concentrate copper for subsequent extracting it by electrolysis or with hydrogen under pressure, are still in the stage of development.

The process of copper extraction with cyanide is also in the stage of experimental tests. The process may prove to be useful for floatation dressing tails containing oxidized copper,

chalcocine and covellite, gold and carbonates inapplicable for LPP process though it will hardly find wide application as well as ammonia leaching requiring special apparatus and increased capital costs.

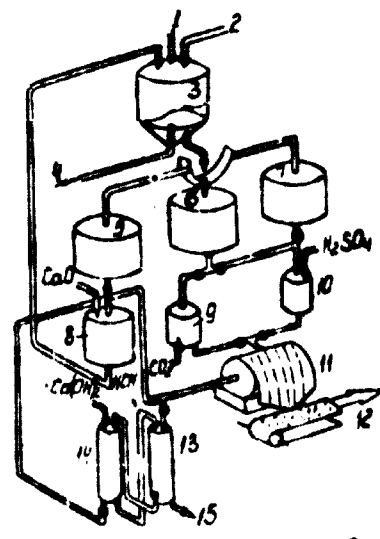


Рис. 1 Схема цепи аппаратов цианидного процесса.

- 1. вода;
- 2. песковая фракция хвостов;
- 3. чан для выщелачивания;
- 4. пески в отвал;
- 5. раствор на рециркуляцию;
- 6. богатый раствор;
- 7. разбавленный раствор;
- 8. раствор в оборот;
- 9. карбонизатор;
- 10. регенерация HCN и осаждение Cu_2S ;
- 11. отделение твердого;
- 12. сульфидный концентрат на переработку;
- 13. две промежуточные башни;
- 14. абсорбция газообразного HCN;
- 15. вода на сброс из 2-ой башни;

Fig 1

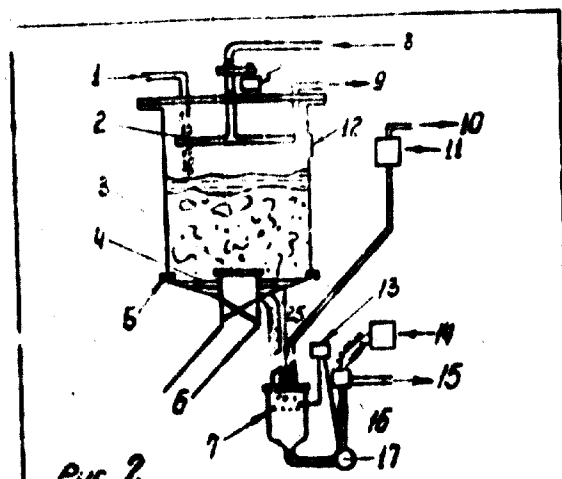


Рис 2

- 1. вход растворителя;
- 2. распределитель песка;
- 3. кольцо для выпуска песка;
- 4. фильтр;
- 5. выпускной кран для песка;
- 6. выводная труба;
- 7. ресивер;
- 8. песковая фракция хвостов;
- 9. к скрубберу;
- 10. к вакуум-насосу;
- 11. вакуумная лобушка;
- 12. смотровое отверстие (окно);
- 13. автоматический контроль уровня;
- 14. датчик показаний электропроводности;
- 15. вывод отработанной раствора;
- 16. проба на электропроводность;
- 17. насос.

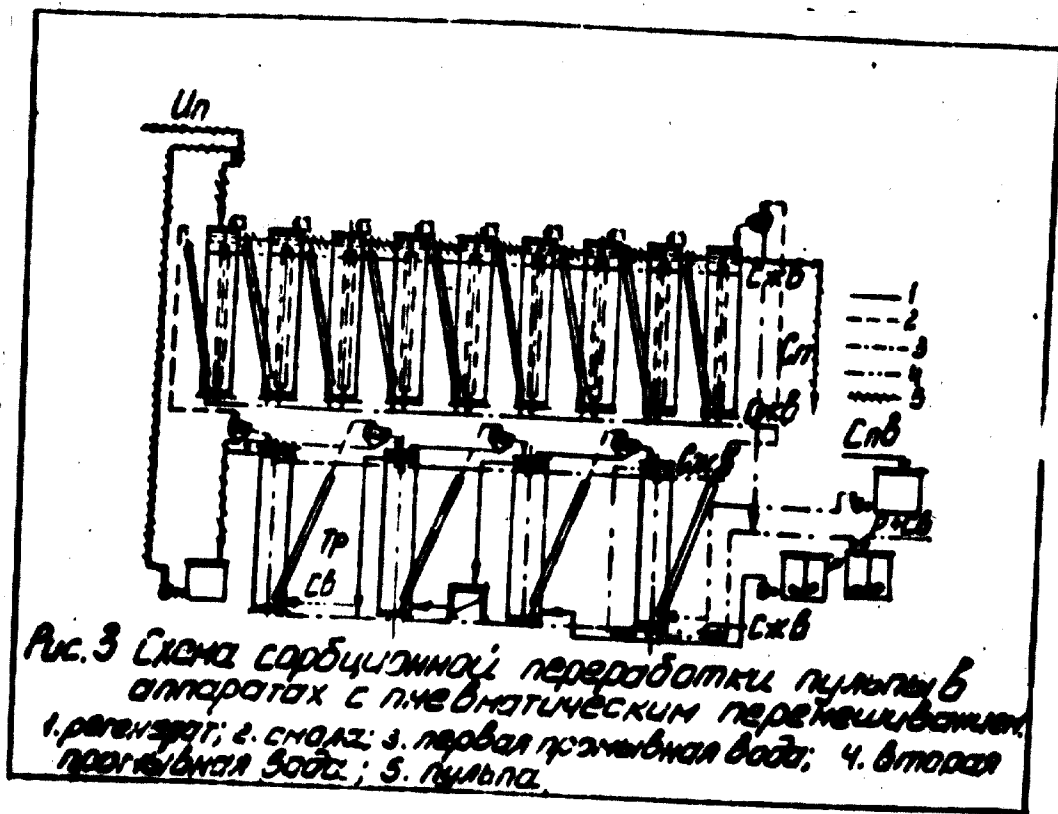


Рис. 3 Схема сорбционной переработки пыли в аппаратах с пневматическим перемешиванием.
1. регенерат; 2. смола; 3. первая фронтальная вода; 4. вторая фронтальная вода; 5. пыльца.

Fig. 3.

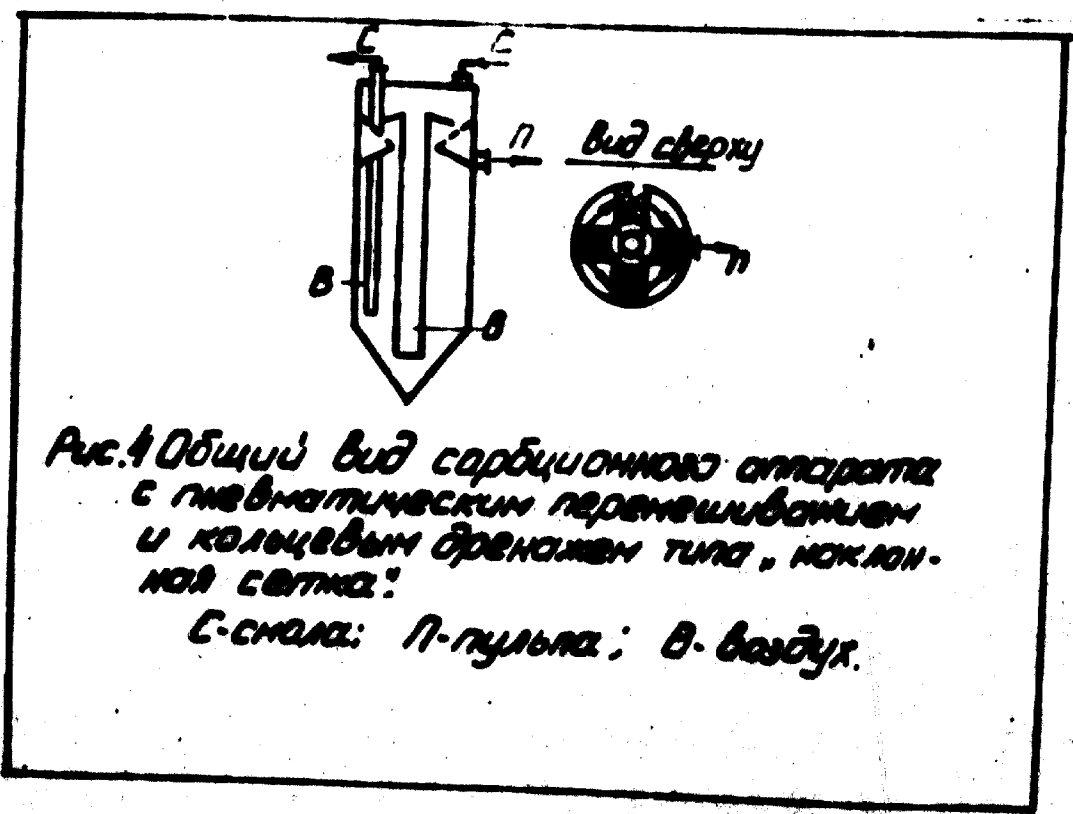


Рис. 4 Общий вид сорбционного аппарата с пневматическим перемешиванием и кольцевым дренажем типа «ночная сетка».
С-смола; П-пыльца; В-воздух.

Fig. 4.

Inscriptions to figures

Fig. 1. Scheme of cyanide process apparatus chain.

1. water
2. sand fraction of tails
3. leaching vat
4. sands into dump
5. solution for recirculation
6. rich solution
7. diluted solution
8. solution for recirculation
9. carbonator
10. HCN regeneration and Cu_2S settling
11. separation of solid
12. sulphide concentrate
13. two intermediate towers
14. gaseous HCN absorption
15. water for discharge from 2nd tower

Fig. 2

1. solvent input
2. sand distributor
3. sand discharge
4. filter
5. discharge cock for sand
6. slime discharge
7. receiver
8. sand fraction of tails
9. to scrubber

10. to vacuum pump
11. vacuum trap
12. watch window
13. automatic level control
14. recording electric conductivity readings
15. waste solution discharge
16. test of electric conductivity
17. pump

Fig. 3. Scheme of pulp sorption treatment in apparatus with pneumatic mixing

1. regenerate
2. resin
3. first washing water
4. second washing water
5. pulp

Fig. 4. General view of sorption apparatus with pneumatic mixing and ring drain of "inclined screen" type

- 0 - resin
П - pulp
B - air

(view from above)

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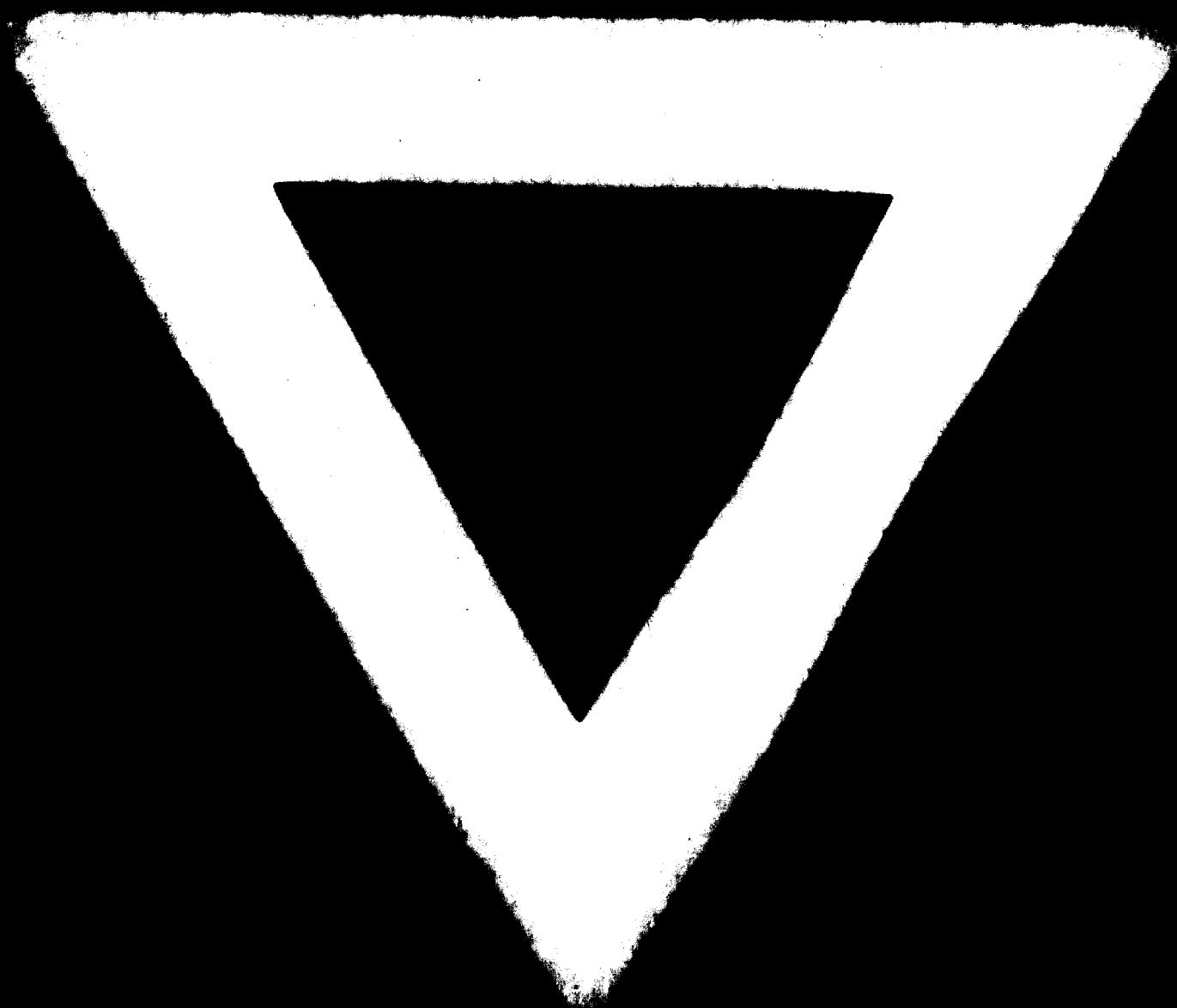
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