HYDROCOPPER® – A NEW METHOD FOR TREATING COPPER CONCENTRATES

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Abstract

Outokumpu has developed a new chloride leach process, called HydroCopper®, to treat copper concentrates. Chalcopyrite is quite refractory in leaching and in sulfate milieu an autoclave of high temperature and pressure is required. However, in the HydroCopper® process copper sulfides can effectively be leached in atmospheric pressure using strong, aggressive chloride solution and copper (II) ions as oxidant. Copper is precipitated from the purified pregnant leach solution as copper (I) oxide, which is filtered and reduced by hydrogen gas to metallic copper powder, which is melted and cast into copper product. Sodium chloride formed in precipitation is decomposed in a chlor-alkali electrolyser, the products of which are circulated back to the process: sodium hydroxide to copper (I) oxide precipitation, chlorine gas to leaching and hydrogen gas to the reduction of copper (I) oxide.

During 2003-2004 the performance of the HydroCopper®-process was confirmed in a demonstration plant processing one ton of copper per day. It has been found to be applicable for different kinds of concentrates and can also tolerate a wide range of impurities. A significant advantage of the process is that gold can be leached as a chloro-complex and recovered with active carbon.

HydroCopper® can economically recover copper from concentrates and process to a cast copper product. The best utilization of the process is in most cases achieved when the plant is situated at the mine site. Higher recoveries are attained by the optimization of the whole operation chain from the mine and the concentrator to the HydroCopper® plant.
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INTRODUCTION

Since the 1970’s, there have been many new hydrometallurgical processes developed for the treatment of copper sulphide ores and concentrates. Almost all of these have included solvent extraction (SX) and/or electrowinning (EW) in the process route to produce a cathode or powder product.

Outokumpu Technology has developed a unique process called HydroCopper® that does not use SX or EW but produces a high purity and high value oxygen free copper product that may be sold directly to manufacturers or to the market.

This process allows mining companies to take control of the complete “mine to market” process route, bypass the custom smelters and rod companies and realize the benefits of the downstream added value process steps.

When the HydroCopper® process is combined with the concentrator at the mine site, the process may be optimized to maximize copper recoveries.

OUTOKUMPU HYDROCOPPER® PROCESS DESCRIPTION

The HydroCopper® process (figure 1) consists of four steps to produce copper powder; leaching of copper concentrate, solution purification, precipitation of cuprous oxide and reduction to copper powder.

Figure 1. HydroCopper® process

The lixiviant (sodium chloride) is regenerated in a chlor-alkali electrolyser cell and oxygen free high purity copper wire rod or any other cast copper product is produced via an induction furnace and a copper upcasting machine (Haavanlammi, Hyväri, Yliö, 2005).

Leaching

Leaching takes place in agitated reactors at atmospheric pressure and temperature of 85 –100 deg C. Three countercurrent stages are required to leach chalcopyrite concentrate, with a thickener after each stage for liquid/solid separation. Total retention time in copper leaching varies between 15 – 25 hours. The most important factor in leaching is the mineralogy of the raw material used. Not only can the copper minerals’ leaching rates differ; gangue materials and iron mineralogy will also have an influence on leaching.

Concentrate, a strong sodium chloride solution and air or oxygen are used in all the leaching stages to generate a strongly oxidizing environment for the copper and gold in concentrate to leach as effectively as possible.

In the first stage fresh concentrate is mixed with the leaching solution from the second stage of leaching. In this stage generally 15 – 30% of the copper will be leached. In the
second stage the leaching pH is controlled to a level of 2.0 – 2.4 by air or oxygen to leach the majority of the copper. The third leaching stage is also oxidative and will leach the remaining 2 – 5% of the copper in concentrate. Copper yield in leaching has been at least 98% for every concentrate tested.

If gold is present in the concentrate, it starts to leach after most of the copper in concentrate has been leached. Gold leaches, when the redox-potential of the leaching solution is raised to a 650 mV (Ag/AgCl) level. The leaching rate and efficiency depend greatly on the mineralogy of the concentrate.

The final leach residue is filtered out of the underflow from the thickener after the third leaching stage. The leach residue consists primarily of elemental sulfur and hematite.

**Solution Purification**

Solution purification is carried out in four stages; cupric precipitation, silver removal, metal precipitation and ion exchange.

**Cupric Precipitation**

In the first stage of solution purification, the pregnant leach solution is treated with limestone to remove sulfate - formed from sulfur in leaching - as gypsum, and caustic soda at pH 4 to precipitate the remaining cupric as cupric hydroxychloride called atacamite. The precipitate is circulated back to leaching, where gypsum reports to the leaching residue. Atacamite is re-leached and thus copper losses to the residue are avoided.

**Silver Removal**

In the second stage of solution purification, metallic copper powder and some mercury solution is added to fluid bed type reactors. The copper ensures that any remaining cupric ions are reduced to cuprous and silver cements onto the copper forming a silver-mercury amalgam. When this reaction is complete, the solution (containing less than 1 mg/L silver) is taken to the next solution purification stage.

The silver-mercury amalgam is treated with an oxidative leaching process. Silver is recovered as silver chloride and the mercury chloride solution is circulated back to the silver removal stage.

**Metal Precipitation**

In the third stage of solution purification, sodium carbonate is added and most of the metals (Zn, Pb Ni etc.,) are precipitated as carbonates at pH 6 – 6.5. The main controlling factor in this stage of the process is pH. By careful control pH can be kept low enough not to precipitate any copper from the leaching solution.

**Ion Exchange**

In order to achieve a high purity oxygen free copper product, the final solution purification must be by ion exchange. This takes out the remaining metallic impurities in addition to calcium and magnesium.
The resin used is a selective chelating type cation exchange resin. The weak elution acids used in the ion exchange are circulated either back to the process or the acid fractions can be separated to allow an impurity bleed from the process.

Precipitation of Cuprous Oxide

The third step of the process is the precipitation of cuprous oxide. Sodium hydroxide liquor from the chlor-alkali electrolytic cell precipitates the cuprous oxide as powder at pH 9 - 10. The slurry is washed and filtered on a vacuum belt filter while the filtrate, sodium chloride solution, is sent to the chlor-alkali cell.

Reduction to Copper Powder

Cuprous oxide from the precipitation step is then fed to a belt furnace with temperatures in the range of 650 – 850 deg C and with hydrogen-nitrogen flow. In this stage the oxygen content is reduced to a level of 200 – 300 ppm and the copper powder discharged, having been slightly sintered.

Casting a Copper Product

The reduced copper plates from the belt furnace can be converted to a high purity oxygen free copper product such as slabs, strips, billet or rod via an induction furnace. The furnace has a graphite layer on top to remove the remaining oxygen (figure 2).

Figure 2. Graphite layer on top of the copper melt in the induction furnace

Regeneration of Chemicals

A key feature of the HydroCopper® process is the use of chlor-alkali electrolysis for the regeneration of chemicals. The sodium chloride filtrate generated at the precipitation of cuprous oxide is fed to the chlor-alkali electrolysis cell, where the salt is decomposed by electricity to sodium hydroxide, chlorine and hydrogen.

The sodium hydroxide is re-circulated to the precipitation of cuprous oxide, the chlorine gas is used in the leaching and the hydrogen gas is used in the reduction to copper powder.

DEMONSTRATION OF THE OUTOKUMPU HYDROCOPPER® PROCESS

Outokumpu has built a demonstration plant with a capacity of one tonne per day copper at our research centre in Pori, Finland. The plant operated during 2003 (Leimala et al, 2003) and 2004 with test campaigns of two different copper concentrates. Laboratory testing is continuing for different concentrates with approximately 15 different copper concentrates tested to date.

The demonstration plant was constructed from industrial grade equipment to test and confirm the materials of construction.
Feed Material to the Demonstration Plant

The feed material to the demonstration plant was selected to be Pyhäsalmi copper concentrate (table 1) from the Inmet-owned mine in Central Finland. The selection was based on the fact that chalcopyrite concentrate is the most difficult copper concentrate to leach, even in the aggressive chloride environment. Also the soluble metallic impurities were high, which gave a good challenge for the solution purification part as well.

Table 1. Pyhäsalmi concentrate; chemical analysis and mineralogy

<table>
<thead>
<tr>
<th>ANALYSIS</th>
<th>(%)</th>
<th>Mineralogy</th>
<th>(%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu</td>
<td>25.73</td>
<td>chalcopyrite</td>
<td>74.00</td>
</tr>
<tr>
<td>Fe</td>
<td>28.11</td>
<td>pyrite</td>
<td>9.26</td>
</tr>
<tr>
<td>Zn</td>
<td>2.09</td>
<td>pyrrhotite</td>
<td>0.36</td>
</tr>
<tr>
<td>As</td>
<td>0.02</td>
<td>sphalerite</td>
<td>3.55</td>
</tr>
<tr>
<td>Pb</td>
<td>0.73</td>
<td>galena</td>
<td>0.86</td>
</tr>
<tr>
<td>S</td>
<td>32.29</td>
<td>others</td>
<td>11.97</td>
</tr>
<tr>
<td>Au g/t</td>
<td>6.00</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ag g/t</td>
<td>240.00</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

No extra grinding has been found necessary, although the concentrate was rather coarse.

Equipment Requirements

Strong chloride environments are demanding for most of the structural materials and there is always concern for maintenance. With this in mind the selection of suitable materials of construction is very important. The leaching and solution purification reactors used in the process are all Outokumpu in-house design.

All the reactors, tanks and thickeners are manufactured entirely of FRP (Fibreglass Reinforced Plastic). FRP is a composite structure consisting of laminated fibreglass and vinyl ester resin in multiple layers. Resin acts as glue binding together glass fibre threads, which give the structural strength to the equipment. Resin enables FRP’s excellent resistant to variety of chemicals.

The reactor’s cylindrical part is usually fabricated on a winding machine where fibreglass threads and resin with hardener are wound onto the outer surface of a cylindrical mould. The reactor top and bottom are fabricated on a special mould by applying layers of fibreglass mat and resin by hand. When required structural thickness is achieved the FRP element is detached from the mould. The elements are joined together to complete the equipment and the mould can be reused to make another element.

Agitators are made of standard structural steel, which is coated with either FRP or special rubber depending on application. Leaching and copper (I) oxide precipitation reactors are temperature controlled with an inner piping system made of titanium.

There are two different types of pumps in the process: centrifuge or hose type with plastic PVDF or PTFE linings. Where any metal is in contact with chlorine, it is selected to be titanium. The demonstration plant is fully automated and instrumentation was at a level of a
commercial scale plant. Different types of meters and other devices were tested and the best were selected for the commercial plant.

All the equipment at the demonstration plant has been commercial type equipment, which has helped to see and overcome most of the material-related questions that the process has faced. The demonstration plant operated for a period of nine months in 2003 and more than six months in 2004. There was a break of several months between test campaigns. This allowed the equipment and material selections to be checked and proved an excellent test of endurance. The materials for a commercial scale plant have been tested and the final selection has a strong information database to support it.

EQUIPMENT USED AT THE DEMONSTRATION PLANT

The following technologies and equipment of Outokumpu are included in the HydroCopper® plant:

- Supaflo® thickeners and Leaching OKTOP™ reactor
- SepTor Ion exchanger
- Proscon® process control system and Courier® continuous analyzer

OPERATIONAL RESULTS FROM THE DEMONSTRATION PLANT

The goal for the leaching part of the HydroCopper® process has been a 98% copper leaching yield and 90-95% gold leaching yield. The rest of the HydroCopper® process—namely solution purification, copper oxide precipitation, copper reduction and copper rod making—accounts for another 2% loss, the total yield of the process being at 96% level.

Leaching

The leaching part of the process has proven itself in leaching chalcopyritic copper concentrates. The copper leaching yield in the demonstration plant has been 98% throughout the balanced period of the operations, which lasted 8 weeks. Total yield for copper has been 96% for the process from concentrate to rod. Other copper sulphide minerals are leached more easily than chalcopyrite, which gives room to optimize the yield even higher.

Leaching and solution purification reactors were designed at Outokumpu Technology in Espoo, Finland. The model used in this type of processes is called OKTOP™ reactor. The solid-liquid separation was operated with Outokumpu Supaflo® high-rate thickeners.

The size of the reactors at the demonstration plant varies from 1 m³ to 10 m³. The biggest reactors are required in the leaching and oxidation parts of the plant. When a lot of heavy solids was present, the mixer wear rate was a great concern. All the mixers in the leaching reactors were alike: a double blade, with an intensive gas dispersing blade in the bottom and an A-model blade on top.

During the first operation the first leaching reactor that takes the heavy concentrate had a significant wear problem in the mixer, but that was due to the coating cracking at the end of the blades. This allowed the strong saline solution to enter the steel core, which did not last very long (figure 3).
Solution Purification

There was a 2% copper loss in solution purification after leaching. Some copper was precipitated into the carbonate precipitate, which was the major proportion of the 2% loss. A Hoesch filter press is used in the carbonate residue filtering (figure 4). The residue was selected to be carbonate instead of hydroxide, because carbonate residue is filtered more easily.

Figure 4. Hoesch filter press in carbonate precipitation stage

The silver removal at the demonstration plant was operated with very fine copper powder. There were some difficulties seen in floating of the powder from one reactor to another, because the powder took the already formed amalgam with it. However, the cartridge filters at the end of the process stage were able to prevent mercury from bleeding out of the process stage. The silver removal reactors in the commercial scale plant will be fluid bed type of reactors, which have been found reliable in other process options of similar powder-solution applications.

The ion exchange unit is a carousel type unit, where all incoming streams may be adjusted according to impurity concentrations in the feed solution. It is easy to control since each stream can be adjusted individually, and chemical consumption can be adjusted to the process solution. The Courier on-line analyzer was a great help in operating the SepTor unit. When the feed solution analysis is updated to the process control screen every half an hour, the feed streams to the carousel can be adjusted to suit. In case there is a breakthrough of metals in the ion exchange process, the on-line analyzer can also detect that from the outlet solution of the SepTor carousel. The pH level is also a strong indication of the quality of the feed solution, since the resin has full capacity close to neutral pH, 6 - 7. If the feed solution pH is much lower, it usually indicates problems in the previous process steps.

Copper (I) Oxide Precipitation

Figure 5. Copper product analysis and copper (I) particle size analysis in November 2003

The copper (I) oxide particle size corresponds very strongly with its purity. For example metals breakthrough due to heavy loading on Nov 13th to 15th can be seen in figure 5. At the same time also the product analyses show quite low concentrations for zinc and lead (both below 5 ppm).

Copper (I) oxide powder has to be washed thoroughly because of product purity. Also the filter must be constructed of materials that do not corrode. Oxidation of the copper (I) oxide powder is not as fast as thought before; the filter can be operated in normal, air atmosphere. No nitrogen protection is needed.

Copper Production

In the demonstration plant Upcast® technology was used to produce wire rod, a product with greater added value compared to copper cathode. Here, the cast wire rod diameter can be adjusted between 8 – 16 mm depending on the nozzle size of the Upcast machine. In commercial applications, a larger range of diameters may be selected.
Copper rod was produced in both of the demonstration operations. The product quality from the demonstration plant has proved to comfortably meet the purity requirements for LME grade 'A' copper, which in turn gives confidence to the HydroCopper® purification circuit and its performance.

**Chlor-Alkali Plant**

A major concern for the operation was the quality of the sodium chloride at the inlet to the chlor-alkali cell, because the cell manufacturers set strict quality requirements for the feed.

The advantage of the HydroCopper® process is that the brine concentration is naturally set at the required concentration and the solution purification and copper (I) oxide precipitation are designed to remove the impurities down to trace levels in the brine feed. In addition the cell is safeguarded with its own ion exchange to ensure that the last traces of calcium and magnesium and copper (II) are removed. After the test campaigns no signs of copper contamination were evident in the cell.

**Leaching Residue and Wastewaters**

The leaching residue was filtered on the Pannevis belt filter with a three-stage wash set-up. In table 2 the average analyses of the leaching residue from the demonstration plant are shown. The moisture of the residue was 20%. Most of the copper in the residue is acid soluble, which has back-precipitated from the process. This was further confirmed in an extensive series of laboratory tests.

<table>
<thead>
<tr>
<th>ICP Cu</th>
<th>ICP Fe</th>
<th>ICP Zn</th>
<th>ICP Na</th>
<th>ICP Pb</th>
<th>ICP Mg</th>
<th>ICP Ca</th>
<th>ICP Mo</th>
<th>S (total)</th>
<th>Cl-</th>
<th>Acid soluble Cu</th>
</tr>
</thead>
<tbody>
<tr>
<td>% 1.49</td>
<td>% 23.71</td>
<td>% 0.06</td>
<td>% 2.88</td>
<td>% 0.05</td>
<td>% 3.08</td>
<td>% 0.09</td>
<td>% 31.66</td>
<td>% 3.02</td>
<td>% 0.91</td>
<td></td>
</tr>
</tbody>
</table>

Wastewaters from the plant were treated with sulfide precipitation, and the precipitate was circulated back to leaching. A typical analysis from the wastewater leaving the demonstration plant is given in table 3.

<table>
<thead>
<tr>
<th>ICP Cu</th>
<th>ICP Fe</th>
<th>ICP Zn</th>
<th>ICP Na</th>
</tr>
</thead>
<tbody>
<tr>
<td>mg/l 0.83</td>
<td>mg/l 0.16</td>
<td>mg/l 1.59</td>
<td>g/l 43.12</td>
</tr>
</tbody>
</table>

All waters run out of the plant were settled and afterwards let to the Kokemäenjoki River in the middle of Pori town.

**Filtering of the products and residues**

Pannevis belt filters were used for leaching residue (figure 6) and copper (I) oxide powder filtering. The filterabilities of the solids were very good, better than expected after the
laboratory experiments. The belt filter application was selected for the product and residue filtering, because in most cases they both need very intensive, multi-stage washing. In both of the solids the chloride content has to be washed down in order to avoid NaCl losses.

Figure 6. Leaching residue filter

**Automation and On-Line Measurements at the Demonstration Plant**

The process control system used in the demonstration plant was selected to be the Outokumpu Proscon\textsuperscript{®} system. The automation level at the plant is at a commercial scale level, which gives users’ experience of the commercial scale plant (figure 7).

Figure 7. Control room at the demonstration plant. Outokumpu Proscon system at hand

The pH measurements are the most valuable indicators of the metal precipitation part of the process. On-line pH values on the process control screen give the operator enough information on each stage of the process in the field. The process is operated with an automated feed of the precipitation chemical. The feed is determined by the pH measurement, which gives the control loop to a chemical feed pump. The automation was found very useful, since chemical consumption was adjusted to the feed solution and was on demand. With a steady feed of the chemical, the consumption is usually not optimized.

pH electrodes were hard to find for a strong chloride process, but thanks to massive laboratory testing programs at Outokumpu Research laboratory, the right kind of electrode was found. In the beginning the pH and redox electrodes were calibrated in every shift, but later on it was found to be unnecessary. The electrodes were cleaned with water and diluted acid, especially in the reactors, where solids were present. The process control system was a valuable tool in following the performance of the electrodes, since the pH values tended to change dramatically, when the electrodes were dirtied. The trendline application of the control system was used to show the operator when the electrode was not working properly.

Some pH and redox electrodes lasted significantly longer than others in the HydroCopper\textsuperscript{®} process. The selection of appropriate electrodes was confirmed during the demonstration plant operations, which was a great help in designing and planning the automation and process control part in the commercial scale plant.

A Courier analyser system was implemented at the demo plant in the first operation. The analyser had 11 sampling points, all of which were fully automated. The solid containing samples were filtered automatically and the solutions were analysed for Cu, Fe, Zn and Pb.

**PROCESS INPUTS**

Many chemicals are used in the HydroCopper\textsuperscript{®} process. Calcium is the only component that has to be purchased outside; all the others can be made within the process itself. The actual input requirements depend on the concentrate composition and are confirmed after tests have been carried out. The outside inputs therefore may be considered broadly as follows:

- power (Electricity) for agitators, pumps, chlor-alkali electrolyser and miscellaneous items;
• calcium compound for solution purification;
• steam for clean condensate supply and for evaporation of excess water;
• carbon if gold is to be recovered

COSTS

The construction and operation of the demonstration plant has enabled the operating costs to be estimated for a variety of concentrate types. It should be noted that the operating costs depend largely on the mineralogy of the concentrate. Concentrates may require two or three leaching stages depending on composition and the soluble impurities have an impact on the quantity of chemicals required.

Estimates for operating costs vary between US10-15c/lb of copper produced; gold leaching and recovery from the leach solution is estimated to add approximately one c/lb of copper produced.

The biggest single cost factor is electricity; this accounts for more than 40% of the overall cost. Therefore the price of power can be an important determinant for a HydroCopper® plant.

One important point is that the chlor-alkali cell may be designed to operate, driven by any one of its products, in a way that is most cost effective for the specific site. For example, if a good local market exists for sodium hydroxide or related products, the cell could be designed to produce excess NaOH for additional revenue.

With regard to plant capital cost, this is a little more complex since not only does the concentrate grade and mineralogy have an impact, so does the plant capacity and local construction costs. As a result, capital costs must be evaluated case-by-case.

ADVANTAGES OF THE HYDROCOPPER® PROCESS

The HydroCopper® process to be generally most economical between the capacities of 40,000 – 200,000 tpa of copper produced.

The HydroCopper® process offers the advantage of being able to process sulphide concentrates. Concentrates with diverse or low copper content and a variety of impurities may be processed allowing lower cut-off grade and increased mine life.

When located adjacent to a concentrator, the process allows significant savings in time and expense compared to transportation of concentrate to a smelter. Transporting secondary gangue materials is unnecessary; only the value added metals such as copper, gold, silver and other products are transported from the plant site.

Working capital requirements are substantially reduced since the concentrate is processed to copper product within days and shipped directly to customers or exchange markets. This allows realisation of revenues months faster than when selling concentrate.

While power consumption is a major cost for the HydroCopper® process, it is nevertheless significantly less than for an equivalent capacity hydrometallurgical process that utilises solvent extraction – electrowinning (SX-EW).
CONCLUSIONS

The Outokumpu HydroCopper® process is a simple way to begin production of high quality copper. When attached to a concentrator at the mine site, the process is an economical way to produce a premium copper product directly saleable to the copper market without influence from the vagaries of the concentrate market.

The process has proved itself in operation at a demonstration plant in Finland operating at a capacity of one tonne per day copper. The process has been operating continuously long enough to prove materials of construction and obtain design parameters for a commercial plant.

Copper miners and producers now have an opportunity to enter the copper product business using the cost efficient Outokumpu HydroCopper® process.
REFERENCE
