

Analysis of the Technology for Obtaining Copper Powder from Technological Solutions of a Copper Plant

Boltaev O.N.

Almalyk branch of Tashkent State Technical University, Almalyk, Uzbekistan

E-mail: boss.olmos@mail.ru

Abstract— In this research work, the technology of extracting copper powder from copper-containing solutions was studied. This paper provides information on the recovery of copper from sulfate solutions using iron scraps and the production of copper powder by electrolysis. The change in particle size when recovering copper powder with iron when the solution is heated from 30 °C to 90 °C was studied, and it was shown that an increase in temperature significantly affects the reduction of particle size. Studies were conducted on the change in current density when obtaining copper powder by electrolysis, and it was observed that when the current density is less than 5 A/m², copper precipitates on the cathode without separating, and when the current density increases to 15-35 A/m², copper is released as powder. In conclusion, both of the above methods are effective in obtaining copper powder from copper-containing technological solutions. Copper powder is more expensive than cathode copper, and its production is economically viable for metallurgical enterprises.

Keywords: technogenic solutions, copper powder, cementation, current density, electrolysis, reducing, iron scraps, decantation.

1. INTRODUCTION

Today, the development of electronic technologies and various electronic equipment requires the use of metals and metal alloys that have no less electrical conductivity, elasticity, and thermal conductivity, but are much cheaper, instead of the precious metals used in them.

The main areas of application of copper nanoparticles are: in powder metallurgy, electronics, mechanical engineering, organic synthesis as a good catalyst. Another important area of application of copper-based metal powders is in chemical power sources [1]. The main requirements for metal powders in these areas are: spherical shape of particles; size of powder particles (typically ranging between 20–60 μm (e.g., 20–40 or 40–60 μm depending on application)); chemical purification of metal powder from additives, including high-level oxides; uniformity of the powder particle composition [2]. Manufacturing products from metal powder instead of casting from metals reduces metal consumption by 60–70%, the number of processing equipment, personnel servicing them, and energy consumption per unit of finished product are also reduced [3]. The selection of a metal powder production method is based on the requirements for the final product and the analysis of the economic cost of the processes that affect the cost of the product (capital expenditure, energy consumption, raw material costs, etc.) [4].

Powders are divided into the following conditional groups depending on the size of their particles:

Table 1

Powder sizes				
No.	Naming	Dimensions		
		nm	μm	mm
1	Nano	<10	<0.01	<0.00001
2	Ultra	10-100	0.01-0.1	0.00001-0.0001
3	Highly dispersed	100-10000	0.1-10	0.0001-0.01
4	Fine	10000-40000	10-40	0.01-0.04
5	Medium	40000-250000	40-250	0.04-0.25
6	Coarse	250000-1 000000	250-1000	0.25-1.0

Technological properties of metal powders include: bulk density; compaction; flowability; compressibility. The physical properties of powders include the shape and size of the powder particles, and the specific surface area.

Areas of application of different grades of copper powders. PMS-B - in the automotive industry; PMA, PM - in aviation, electrical engineering, chemistry, engineering: spare parts, filters for electrical machines and filters for oil purification; PMS-1 - in powder metallurgy: in the production of bearings, alloy tools; PMS-K - in the field of electromechanical engineering: for contacts; PMS-N - in the field of metaloceramics: in non-critical parts [1,5,6].

The article [7] presents a method for obtaining copper powder from CuSO₄-containing solutions. Sodium polyacrylate, Na₂SO₃, and NaBH₄ were added to the solution in a ratio of 3:1:2 to separate copper powder from CuSO₄. Sodium sulfite prevents oxidation of the copper powder, and copper powder with a size of 3-14 nm was obtained.

Copper powders available on the world market have an average particle size in the range of 1-50 μm and a surface area in the range of 0.25-1.2 m^2/g [8]. Copper powder particles obtained by the pneumatic circulation method have a particle size of 600-800 nm and a surface area of 6.2 m^2/g [9]. A common method for producing powders is the electrical discharge (ED) method. The electrical discharge generates a high-intensity pulse current passing through a conductor, resulting in local heating and explosion, as a result of which the metal is dispersed to a size of about 50 nm [8, 9, 10]. The disadvantages of the above method are the preparation of the raw material (thin wire), as well as the need for high vacuum and explosion-proof equipment. Physical-mechanical methods also include pneumocirculation technology. It creates interparticle collisions in the uninfluenced gas furnace flows, which are based on a significant increase in the dispersion of the material due to intensification [9, 10].

It should be noted that the need to melt the source leads to the same significant energy costs for almost all physical and mechanical methods. Using the mechanochemical synthesis method, complex oxides and powders of oxides of dispersed elements with nanocrystalline particle sizes of 30-70 nm are obtained, which, in turn, consist of particles with a size of 1-3 nm [11,12].

2. MATERIALS

Along with the main products, the copper smelting plant of Almalyk MMC JSC also produces copper sulfate. The production of copper sulfate is based on the evaporation-crystallization of copper sulfate solutions, and the increase in the content of other non-ferrous metals in the solution worsens the quality of the resulting product. As a result, the solution contaminated with other metals is removed from the technological process. The scheme of the formation of technological solutions as a result of the processing of copper anodes is presented in Figure 1.

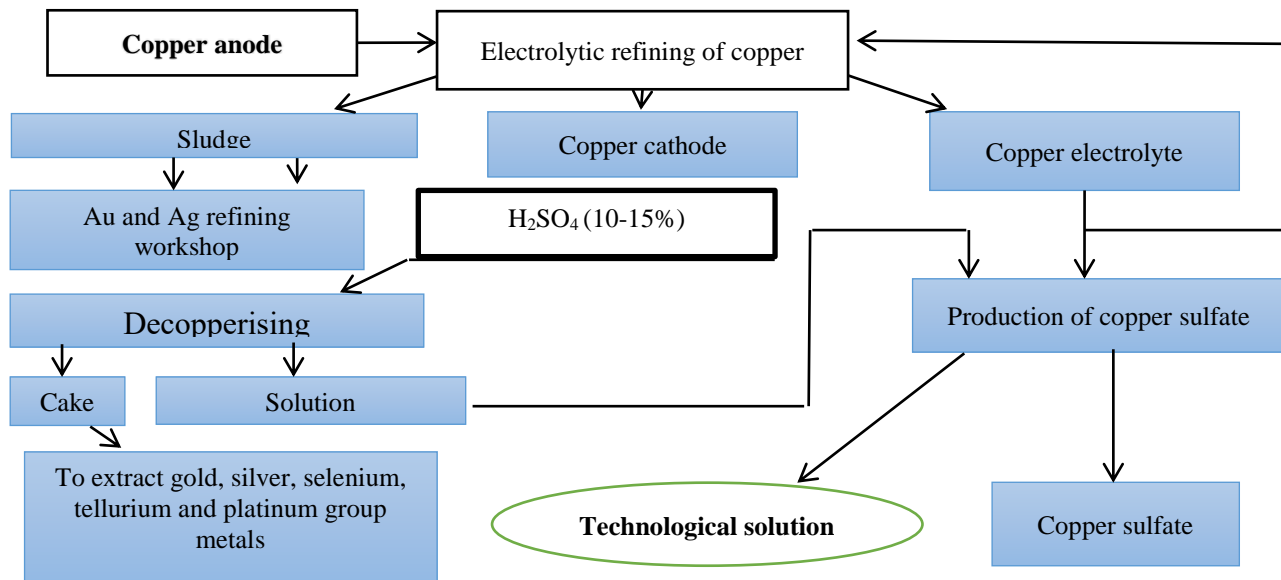


Fig.1. Scheme of the formation of technological solutions

3. RESEARCH METHODOLOGY

Obtaining metal powders by chemical reduction methods [18,19].

Chemical reduction methods are based on the reaction between a metal salt solution and a reducing agent and are carried out at room temperature, elevated temperatures, and pressures [9,13,14]. Borohydride salts, glucose, hydrazine, L-ascorbic acid, and sodium hypophosphite are used as reducing agents [15,16]. The solutions also include additives such as powder particles, fatty acids, and amino acids. Methods for obtaining ultradisperse and nanoparticles using colloidal solutions consist of synthesizing them from the initial reagents in the solution and stopping the reaction at a certain time. transitions from a colloidal liquid state to a solid dispersed phase to a dispersed solid state [14,16,17]. The most common copper powders, with particle sizes of 10–100 nm, are obtained by chemical reduction methods [8, 10]. The production by these methods depends on the volume of solutions used.

Chemical recovery (cementation) and electrolysis methods for obtaining copper powder from solutions were experimentally tested.

When obtaining powder by cementation method, solutions with a copper content of 70 g/l were prepared. The process of precipitation of copper in powder form from solutions was carried out in the temperature range of 30-90 °C with the addition of iron pieces (Fe). During the same time interval, the experiment was carried out depending on the temperature (with a difference of 10

°C). The cementation process was carried out using iron, forming a solution (FeSO_4) and a precipitate (Cu). The solutions were cooled to room temperature, then the liquid and solid phases were separated using a vacuum filter. The resulting copper precipitates were treated with passivators at a temperature of 25-30 °C so that copper would not actively react with oxygen in the air and oxidize. The precipitate was mixed with a passivator (CH_3OCH_3), washed three times in distilled water by decantation, and filtered through a nutch filter.

4. RESULTS AND DISCUSSION

The results of a research study on the recovery of copper powder from sulfated copper solutions with iron filings are presented in Table 2.

Table 2

Dependence of copper powder size on process temperature ($\tau=1.5$ hours)

T/R	Temperature at which the experiment was conducted, oC	Product size and quantity, %			
		<20 μm	20-40 μm	40-100 μm	100< μm
1	30	5	12	18	65
2	40	12	18	25	45
3	50	28	27	26	19
4	60	37	25	20	18
5	70	62	15	20	3
6	80	66	16	14	4
7	90	87	9	2	1

Table 2 shows that the size of the powder particles decreased with increasing temperature from 30 °C to 90 °C. The resulting powder was first washed with sodium hydroxide solution, then with acetone and distilled water, and 80-90% of the particles were copper powders smaller than 20 μm .

A scanning electron microscope (SEM) image of copper powder is shown in Figure 1.

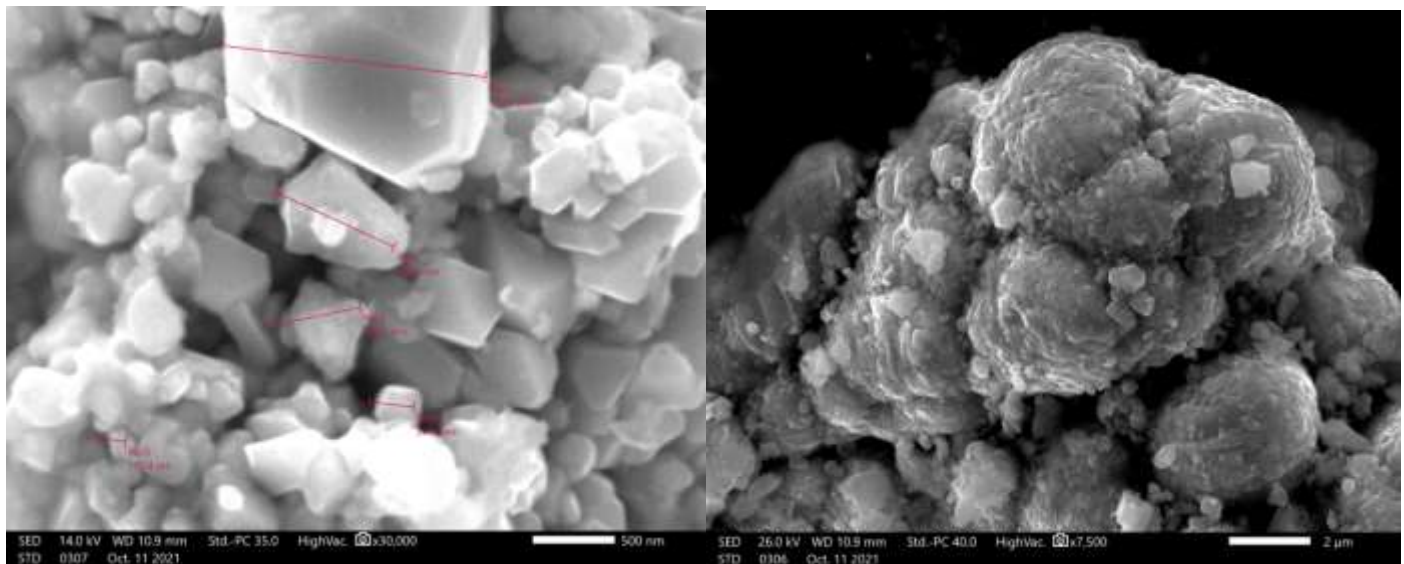


Fig.2. SEM image of copper powder

In the electrolysis method, initially 1 liter of copper sulfate (Cu-50 g/l) solution was prepared at 25-30 °C. The solution was electrolyzed for 180 minutes in a special laboratory electrolyzer.

The experiments were conducted at current densities of 15, 25, and 35 A/cm^2 . The images of the resulting copper powders are shown in Figure 2.

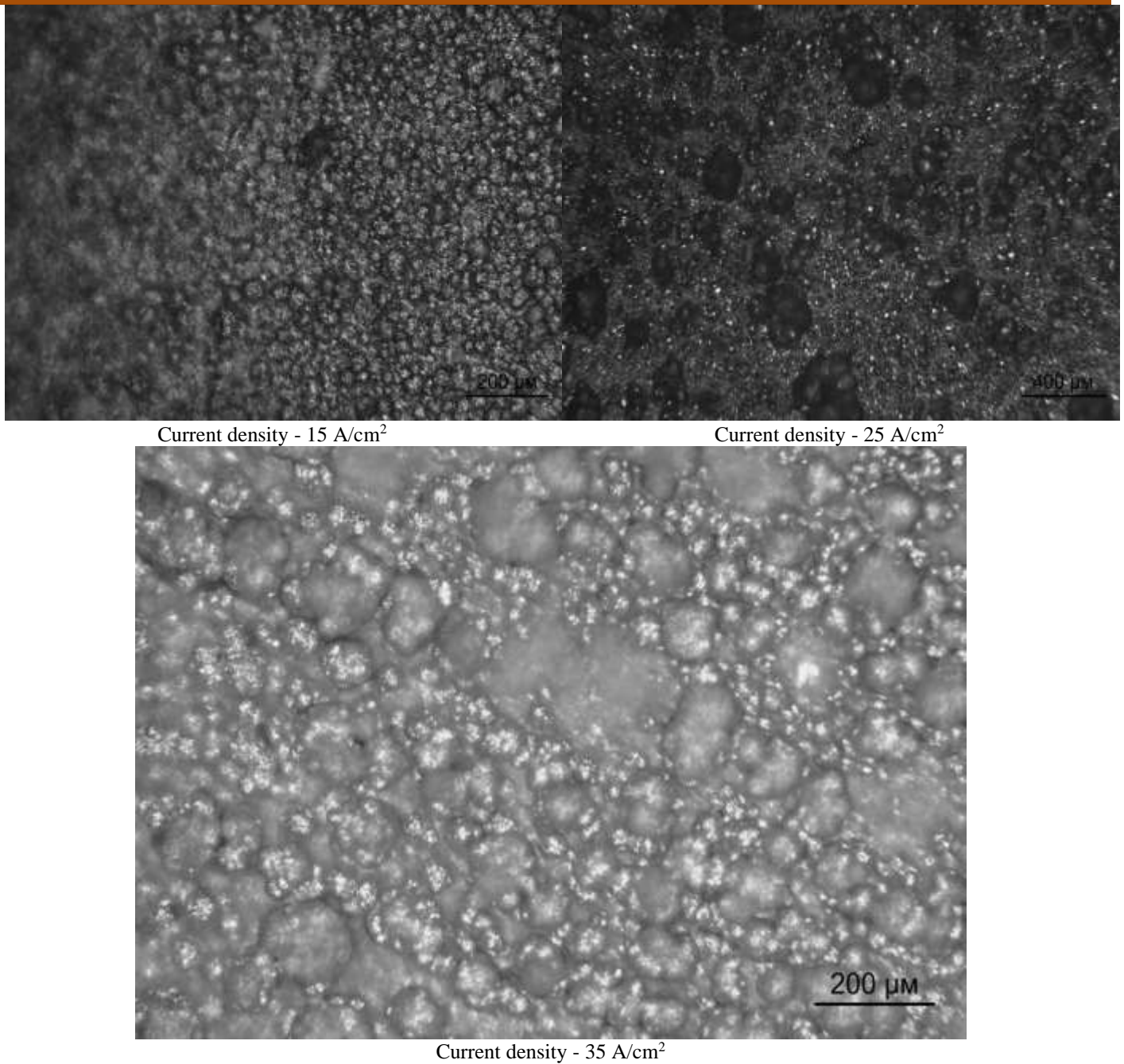


Fig.3. Image of copper powders under a metallographic (Altami MET 3d) microscope

The results show that the size of the obtained copper powders decreased with increasing current density.

The results of the scanning electron microscope (SEM) analysis of the sample of copper powder obtained by electrolysis treated with an organic passivator (CH_3OCH_3) are shown in Figure 3. This figure shows that the powder treated with organic passivators contained carbon in addition to copper in its chemical composition. The powder obtained by washing the powder several times in hot water after the organic substance was removed showed the removal of carbon compounds (Figure 4).

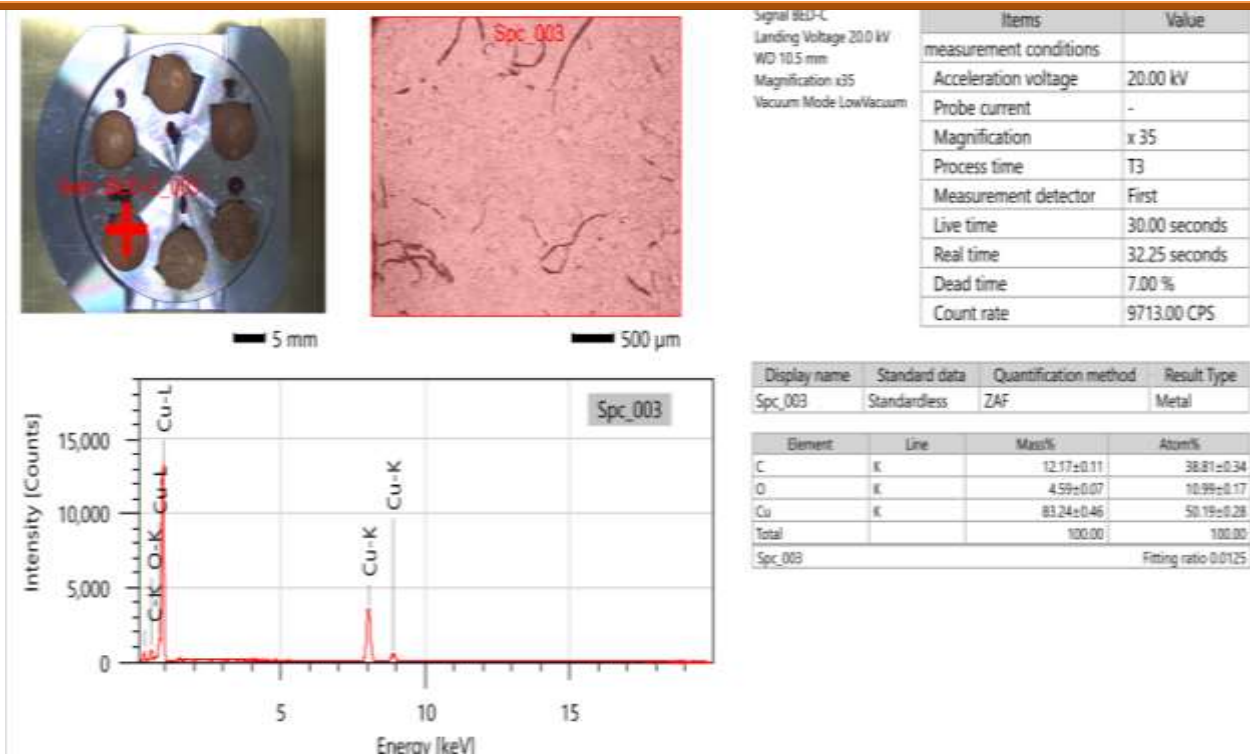


Fig.4. Scanning electron microscope (SEM) image of a sample obtained by electrolysis

Analyzed result (FP method)

No.	Component	Result	Unit	Stat. Err.	LLD	LLQ
1	Cl	0.100	mass%	0.0006	0.0002	0.0005
2	Al	0.125	mass%	0.0035	0.0025	0.0076
3	Si	0.194	mass%	0.0024	0.0008	0.0023
4	S	0.0751	mass%	0.0007	0.0002	0.0005
5	Ca	0.0395	mass%	0.0017	0.0031	0.0093
6	Ti	0.0085	mass%	0.0006	0.0013	0.0038
7	V	0.0035	mass%	0.0003	0.0007	0.0021
8	Cr	0.0304	mass%	0.0005	0.0005	0.0015
9	Fe	1.19	mass%	0.0078	0.0073	0.0219
10	Co	(0.0100)	mass%	0.0015	0.0044	0.0131
11	Ni	(0.0120)	mass%	0.0023	0.0065	0.0194
12	Cu	98.1	mass%	0.0264	0.0004	0.0013
13	Ag	0.0046	mass%	0.0007	0.0007	0.0021
14	Sn	0.0113	mass%	0.0009	0.0009	0.0028
15	Sb	0.0130	mass%	0.0011	0.0010	0.0031
16	Dy	(0.0357)	mass%	0.0043	0.0124	0.0372

Spectrum

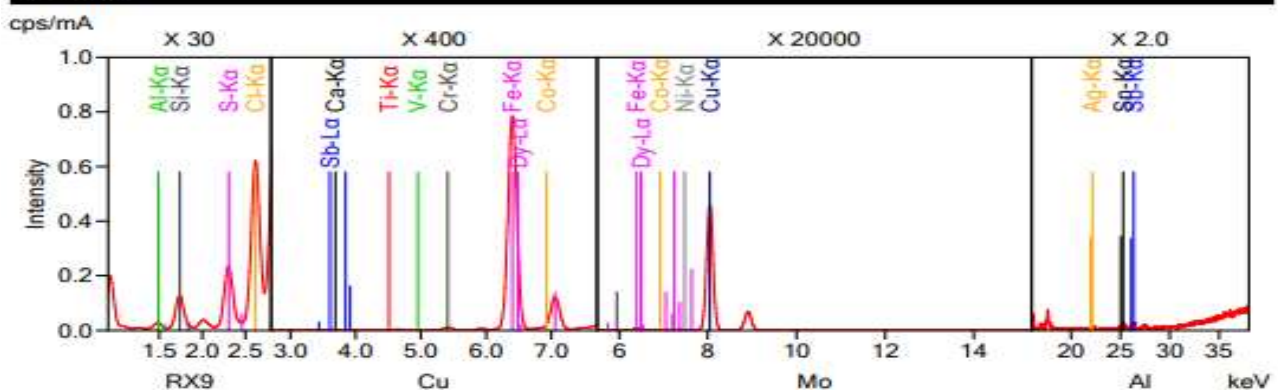


Fig.5. Analysis of a sample obtained by electrolysis on a Rigaku EDXRF NEXCG spectrometer

5. CONCLUSION

When reducing copper from sulfate solutions with iron filings, increasing the temperature causes the powder particle size to decrease. At 30°C, 5% of the copper powder was smaller than 20 µm, while at 90°C, more than 80% of the copper powder was smaller than 20 µm.

When obtaining copper powders by electrolysis, the size of the powder particles depends on the current density, and the experiments were conducted under conditions of 15-35 A/cm². An increase in current density led to a corresponding decrease in particle size.

Analysis of the copper powder precipitated in the solution and processed with organic passivators showed that the chemical composition contained carbon in addition to copper. To remove these organic substances from the powder, it is necessary to wash it several times in hot water.

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