Behavior of Platinum Metal Concentrates under Autoclave Conditions

O. V. Belousov, N. V. Belousova, A. I. Ryumin, and R. V. Borisov

Abstract—Dressing processes of platinum metal concentrates (CP-1 and CP-2) in acid and alkaline media under autoclave conditions were studied. The optimal recovery conditions (time-, temperature-, and concentration-related) of impurity elements were found for the temperature range under study. It was demonstrated that the predominant changes occur during the first 30–60 min. Use of reducing agents can preclude transfer of noble metals into solution in leaching of impurity elements.

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Improving the technologies aimed to obtain rich selective concentrates of platinum-group metals (PGMs) is a rather topical task because removal of base elements from the refining cycle requires a gross expenditure [1]. In this context, this issue attracts a profound interest and has been reflected in a number of publications [2–11]. Researchers consider both pyrometallurgical methods for obtaining noble metals [2, 3] and those of the hydrometallurgical type [4–11]. A particular position among hydrometallurgical techniques is occupied by autoclave processes.

In modern hydrometallurgy, these methods belong to those most rapidly developing and acquire an increasing importance in processing of raw materials containing nonferrous metals in the stages of ore decomposition and concentration and separation of components. The autoclave techniques not only supplement the conventional pyrometallurgical processes, but also successfully compete with these [5].

A characteristic feature of many processes involving platinum-group metals and compounds of these is the kinetic hindrance. Raising the temperature to 100°C not always can eliminate the kinetic hindrance, and, therefore, performing hydrothermal processes in closed systems (autoclaves) is rather attractive, especially in refining, because it is possible in this case to minimize the residual content of noble metals in concentrate processing solutions.

To apparent advantages of the autoclave techniques can be attributed the intensification of the processes as compared with open systems, zero loss of valuable elements, ecological safety, effective use of reagents, and simple standardization.

Examples of an effective application of autoclave techniques in world’s largest companies were reported in [6–8]. A detailed analysis of the development of the autoclave technology in our country and abroad has been made by the team headed by S.S. Naboichenko [9]. Effective approaches to obtaining highly selective concentrates of platinum metals by autoclave oxidative leaching were suggested in [10, 11].

The goal of our study was to determine the possibility of selectively recovering impurity elements (IEs) from concentrates of platinum-group metals under autoclave conditions.

EXPERIMENTAL

Experiments on recovery of base elements from concentrates were carried out in laboratory titanium auto-
claves with removable fluoroplastic linings whose design was described in detail in [12, 13]. The inner fluoroplastic surface rules out loss of noble metals via cementation. The chemical reagents we used in the study were of no less than analytically pure grade.

The fluoroplastic vessel-insert was charged with weighed portions of the concentrate and a sulfuric acid (2 M) or sodium hydroxide (6 M) solution, if not otherwise stated. The role of a reducing agent was played, depending on the medium, by sodium sulfite or sodium formate, respectively. The phases (sediment–solution) were separated by filtration through a Blue ribbon filter; no problems with phase separation were encountered even when alkaline solutions were used. The sediment was dried to constant weight, and then the degree of concentrate dissolution was determined. The chemical composition of the solution was analyzed by atomic-emission spectroscopy with inductively coupled plasma (AES with ICP) on a Spectro CIROS spectrometer (Germany). The phase composition of the concentrates was determined by X-ray phase analysis on a PANalytical X’Pert PRO MPD powder spectrometer.

We used the typical concentrates CP-1 and CP-2 whose chemical composition is presented in Tables 1, 2.

According to X-ray data, the phase composition of CP-1 is mainly constituted by a palladium-based substitution solid solution with gold and platinum incorporated into the crystal lattice; the main peaks are strongly broadened, which points to the chemical inhomogeneity of the solid solutions of the main phase, on the one hand, and to the small size of crystallite, on the other. In addition to the main phase, there is certainly the silver sulfate base. The presence of water-soluble phases is also confirmed by the fact that silver, copper, and nickel appear in solution when the concentrates are brought in contact with water at 25°C. No other precious metals were found in the aqueous solution in this case.

Preliminarily, we examined the effect of temperature on the behavior of CP-1 in 2 N H₂SO₄ at a s : l = 1 : 40 (Fig. 1). Experiments were performed at 120, 150, and 180°C. The 15–18% decrease in the mass of concentrate CP-1 at 180°C is due to the dissolution of As, Cu, Fe, Ni, and Ag. An analysis of the solution demonstrated that the highest concentration is observed in the case of silver, up to 2 g L⁻¹, which corresponds to its 60% recovery. It should be noted that the key role in silver oxidation is played by the atmospheric oxygen from the gas phase of the autoclave.

When the temperature was lowered to 120°C, the parameters of leaching of impurity elements were markedly impaired. At 180°C, a larger degree of leaching was observed (Fig. 1, curve 1), with the main change in mass reached during the first 30–60 min and making the experiment longer not leading to a noticeable transfer of components into solution. Obtaining close results at 120°C (Fig. 1, curve 2) requires no less than 2–4 h. Intermediate results were obtained at 150°C.

Closely similar dependences were obtained with CP-2. In addition, it was found that the main transformation of concentrates CP-1 and CP-2 also occur in aqueous solutions of sodium hydroxide at 180°C during the first 30–60 min. Figure 2 shows how temperature affects the degree of CP-1 leaching with an aqueous solution of sodium hydroxide (6 M).

Further studies were carried out at the technologically reasonable s : l ratio of 1 : 4. In this case, the fraction of

### Table 1. Content of noble metals in the concentrates

<table>
<thead>
<tr>
<th>Concentrate</th>
<th>Pt</th>
<th>Pd</th>
<th>Rh</th>
<th>Ir</th>
<th>Ru</th>
<th>Au</th>
<th>Ag</th>
</tr>
</thead>
<tbody>
<tr>
<td>CP-1</td>
<td>13.3</td>
<td>48.0</td>
<td>0.6</td>
<td>0.069</td>
<td>0.20</td>
<td>2.65</td>
<td>15.4</td>
</tr>
<tr>
<td>CP-2</td>
<td>6.18</td>
<td>25.9</td>
<td>2.0</td>
<td>0.072</td>
<td>0.58</td>
<td>1.31</td>
<td>31.5</td>
</tr>
</tbody>
</table>

### Table 2. Content of base elements in the concentrates

<table>
<thead>
<tr>
<th>Concentrate</th>
<th>Te</th>
<th>As</th>
<th>Cu</th>
<th>Fe</th>
<th>Ni</th>
<th>Pb</th>
<th>Sb</th>
<th>Se</th>
</tr>
</thead>
<tbody>
<tr>
<td>CP-1</td>
<td>2.06</td>
<td>0.67</td>
<td>3.58</td>
<td>0.39</td>
<td>0.83</td>
<td>1.9</td>
<td>0.57</td>
<td>2.76</td>
</tr>
<tr>
<td>CP-2</td>
<td>2.57</td>
<td>2.00</td>
<td>3.3</td>
<td>0.17</td>
<td>0.59</td>
<td>2.9</td>
<td>0.54</td>
<td>7.19</td>
</tr>
</tbody>
</table>