Tailing waste is heterogeneous material, with a great variation in chemical and physical properties. There is a need for its characterization for the purpose of valuable metal recycling, but also for its environmental impact assessment. The material properties of the tailing waste deposit Gornje Polje, like grain size, mineralogy, or even location within the heterogeneous content of the deposits are essential for the decision on the valorization of the large quantity of minerals, some 12,000,000 m³ situated on 500,000 m² of the river bank. In order to propose the treatment process, the material was characterized by instrumental methods of Scanning Electronic Microscopy, X-ray Diffractometry, Atomic Absorption Spectrophotometer and Differential Thermal Analysis. The obtained results have shown that most of lead and arsenic are concentrated in the larger coarse grains in the oldest part of the tailing in the form of oxides, sulfates and carbonates. The grains smaller than 1 mm makes the most of the tailing with 93.07 %, and those are consisted of sulphates: PbSO₄(in the interval from 5.86 to 13.04 wt%), CaSO₄(5.91–75.84 wt%), depending on the stage of transformation of the samples, and sulphides: FeS (2.73-11.42 wt%) and AsS (7.89 wt%). The grains which are larger than 1 mm contain up to 61.76 wt% of PbSO₄ and 32.33 wt% of carbonates-PbCO₃, after the thermal transformation at 350 °C.