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粗$TiCl_4$铜丝塔除钒废水沉淀泥浆综合回收工艺

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摘 要：介绍了一种粗$TiCl_4$铜丝塔除钒废水沉淀泥浆综合回收新工艺。该工艺由沉淀泥浆自氧化、碱洗脱氯、脱氯渣一次酸浸生产硫酸铜、一次酸浸渣苏打焙烧提钒和提钒渣二次酸浸5个主要工序组成。实验结果表明，粗$TiCl_4$铜丝塔除钒废水沉淀泥浆在空气中能自氧化。沉淀泥浆在空气中堆放1个月，接近90%的金属铜变成$CuCl_2 \cdot 2H_2O$，$Cu_2Cl(OH)_3$和$Cu_2(OH)_3Cl$；这些铜的氯氧化合物在碱性溶液中容易转化成$Cu(OH)_2$；在控制液固比4l:，pH值为 11，温度为80 ℃的条件下搅拌1 h，转化率达96%。当酸浸液的pH值为2.0~2.5时，Fe、V、Ti等杂质留在渣中，浸出液蒸发浓缩至密度为1.38 g/cm^3，冷却结晶得到的硫酸铜产品符合国标GB437—93的质量要求。酸浸渣按化学计量的2.5倍加苏打后在700 ℃焙烧3 h，焙烧后按液固比31:加水在70 ℃搅拌1 h浸钒，水浸液按化学计量的3倍加氯化铵沉淀偏钒酸铵，偏钒酸铵在550 ℃热解2 h得到纯度为98.61%的V_2O_5。提钒渣再经二次酸浸。整个工艺过程铜和钒的总回收率分别达到98.63%和95.65%。

关键字：粗$TiCl_4$精制；铜丝；除钒；综合回收

Comprehensive recovery of precipitate of wastewater in removing vanadium from raw $TiCl_4$ with copper-wire

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Abstract: A new method was presented to recover copper and vanadium from the precipitate formed in the waste water after vanadium removal from raw $TiCl_4$ with copper-wire. The recovery process consists of five major procedures, namely, the self-oxidization of precipitate, the removal of chlorine with sodium hydroxide solution, the first-stage leaching of copper with sulphuric acid and recovering vanadium by roasting the first leached residue with sodium carbonate, the leaching with water and the leaching copper with sulphuric acid once again. It is found that the precipitate can self-oxidize in air. After

stacking for one month in air, about 90% metallic copper contained in the precipitate turns into $CuCl_2 \cdot 2H_2O$, $Cu_2Cl(OH)_3$ and $Cu_2(OH)_3Cl$. The copper oxychlorides can easily convert to $Cu(OH)_2$, and the conversion rate is over 96% under liquid-to-solid ratio 41: and pH 11 at 80 ℃ by stirring for 1 h. When pH value is maintained in the range of 2.0−2.5 during the leaching of sulfuric acid, the impurities of Fe, Ti and V are remained in the leached residue. And then the leaching liquor is concentrated to 1.38 g/cm^3 by evaporation and cooled to obtain the product of $CuSO_4 \times 5H_2O$, which is in accordance with the standard of GB437─93. After adding sodium carbonate under the stoichiometric proportion of 2.5 the residue is roasted at 700 ℃ for 3 h. The calcined product is leached with water under liquid-to-solid ratio 31: at 70 ℃ by stirring for 1 h. NH_4Cl is then added in the leaching liquor containing vanadium according to the stoichiometric proportion of 3 to obtain the precipitate of NH_4VO_3. When NH_4VO_3 is thermolysized at 550 ℃for 2 h, V_2O_5 with the purity grade of 98.61% is produced. After vanadium removal, the residue is leached once again with sulfuric acid. The total recoveries of copper and vanadium are 98.63% and 95.65%, respectively.

Key words: raw $TiCl_4$ purifying; copper wire; vanadium removal; comprehensive recovery

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