

## 载Ag (Cu) 凹凸棒石抗菌剂及其性能研究

论文标题:载Ag (Cu) 凹凸棒石抗菌剂及其性能研究

Research of Ag-carrying (Cu-carrying) Attapulgite Antibacterial Agent and Its Properties

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inorganic antibacterial agent, Ag-carrying antibacterial agent, Cu-carrying antibacterial agent, attapulgite, preparation, properties, ion exchange, ion adsorption, antibacterial action

本文对以凹凸棒石为载体的银、铜抗菌剂的制备工艺及性能进行了试验研究。工艺试验研究结果表明: 1、天然凹凸棒石(以下简称凹土)需通过提纯方可作为抗菌剂载体。研究形成的有效提纯方法: 凹土以7: 93土水比浸泡48h后高剪切分散3min, 并以0.4gNaOH / 100g土降粘, 再离心分离。其中提高离心温度可使分离效率提高2~3倍, 通常采用30~40℃的温度。实验室提纯收得率为33%~45%。2、硝酸湿法活化处理可使凹土的载Ag能力从0.17gAg / 100g土提高到0.57gAg / 100g土, 同时大幅度提高凹土的白度。研究得到硝酸湿法活化法较佳工艺条件为: 硝酸浓度0.8mol / l, 活化温度98℃, 活化时间80min。采用硝酸是为了避免在金属加载过程中产生银盐沉淀。3、影响载Ag的主要因素按影响程度大小为: 硝酸银溶液浓度、反应温度、反应时间; 影响载Cu的主要因素按影响程度大小为: 反应温度、反应时间、硝酸铜溶液浓度。总结金属离子加载试验结果, 其较佳合成工艺为: 载银以凹土与0.03mol / l硝酸银1: 20固液比在60℃水浴中处理5h为宜; 载铜以凹土与0.05mol / l硝酸铜以固液比1: 20在90℃水浴中处理6h为佳。性能研究显示: 1、载银和银铜复合抗菌剂对大肠杆菌和金黄色葡萄球菌的抑菌率均为100%, 载铜抗菌剂对两者的抑菌率分别为82.2%, 87.3%; 载银抗菌剂的瞬时抑菌效果优于载铜抗菌剂, 复合抗菌剂的瞬时抑菌性能好过纯载银抗菌剂。2、载银、载铜抗菌剂在静水中时在开始3h内快速释放, 至72h趋于平衡。72h时释出百分比分别为8.55%和7.67%。3、载铜抗菌剂的耐光性较好, 空气中日常光照条件下90天以上未见明显变色。而载银抗菌剂15天以上开始发灰并不断转黑。银铜复合抗菌剂也有不同程度的变色倾向。综合原子吸收光度分析、XRD分析和红外图谱分析认为所合成的载银、载铜凹土抗菌剂中, Ag、Cu是以离子形式存在的, 而载银、载铜凹土抗菌剂仍保持原有的晶体结构, 凹土特有的优异性能(如胶体性、吸附性等)也未有明显改变。DSC分析显示凹土载铜后其结晶水含量明显提高。而载银载铜后结晶水、沸石水和结构水的失去温度略有提高。

Preparation technics and properties research of Ag-carrying antibacterial agents and Cu-carrying antibacterial agents by using attapulgite as carrier were done in this thesis. The results of technics research showed: 1. To be used as antibacterial carrier, crude attapulgite should be purified. An effective method was formed through the researches: First attapulgite and distilled water were mixed in the ratio of 7:93 for 48h, then separated for 3min. 0.4gNaOH/100gclay were used to reduce the viscosity. At last attapulgite were apated by centrifugation. The efficiency was enhanced by increase of the centrifugation temperature. 30-40℃ were used better. And the reclaim-ratio of purification is 33%-45% in lab. 2. The carrying capacity of attapulgite could be enhanced from 0.17gAg/100gclay to 0.57gAg/100g clay afer attapulgite have been activated by HNO3 in waterish. And through the same process attapulgite could be bleached. The better technical conditions were showed by the testings: after have been treated with 0.8mol/l HNO3 in a 98℃ water bath for 80min, attapulgite could get the optimal activation effect. HNO3 was used to avoid creating precipitable Ag-salt. 3. Researchs showed that the primary factors for Ag-carrying process is concentration of AgNO3, reaction temperature, reaction time; and for Cu-carrying process is reaction temperature, reaction time, concentration of Cu(NO3)2. The better process conditions for Ag-carrying is treating the attapulgite with 0.03mol/l AgNO3 in a 60℃ water bath for 5h, and for Cu-carrying is treating attapulgite with 0.05mol/l Cu(NO3)2 in a 90℃ water bath for 6h. Properties research showed: 1. three kinds of antibacterial agents were all of excellent antibacterial actions. The colon bacillus and the golden staphylococcus could be 100% killed by Ag-carrying antibacterial agent and composite antibacterial agent. And the antibacterial-action-rates of Cu-carrying were 87.3% and 82.2%. 2. At the first 3h Ag-carrying and Cu-carrying antibacterial agents were released quickly in stati water. After 72h the releasing came into equilibrium and the percent of releasing were 8.55% and 7.67% respectively. 3. The testings of lightfastness showed that Cu-carring antibacterial agent have the best sunproof property. It hadn't any changs for 90 days in the atmosphere and ordinary sunshine. At the same condition, the color of Ag-carrying antimicrobial began to change from ivory white to light gray at the first 1 5days, then change from light black to black. The color of composite antibacterial agent was changed a little too. And the mechanism and corresponding countermeasures for color-change should be researched ulteriorly. XRD, infrared analysis showed that the crystallinity was reduced by activation, while the full crystalline structure of attapulgite wasn't changed by activation and adsorption. It's adsorbent property and collioidal property were still remained. DSC analysis showed that the crystal water were clearly increased after Cu-carrying. And the dehydrate temperatures of crystal water, zeolite water, frame water were all increased slightly.

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