# Preparation Method of Copper Modified Violet Tungsten Oxide Photocatalyst
# CN 104785275 A

ABSTRACT

The invention discloses a copper modified violet tungsten oxide photocatalyst material. Copper with a proper concentration is doped, and the copper ions can enter crystal lattices of violet tungsten oxide and introduced into defect positions, so as to influence composition of electrons and electron holes, and under the proper concentration achieve the optimal catalysis effect. Meanwhile, the invention discloses a preparation method of the catalyst material. The copper doped violet tungsten oxide powder is synthesized by a solution method, so that the preparation method is short in reaction time and low in reaction triggering temperature; the obtain nanocrystalline catalyst powder is 30-200 nm in diameter and 1-3 microns in length. The copper modified violet tungsten oxide photocatalyst material and the preparation method thereof disclosed by the invention solve the problem that the photocatalytic performance is remarkably improved by the method of appropriate doping to introduce the defect; the prepared photocatalyst powder has the advantages of being fine in crystalline grain, low in cost, high in raw material powder utilization ratio, high in photocatalytic efficiency and the like.

DESCRIPTION

Method for preparing copper tungsten light purple modified catalyst

TECHNICAL FIELD

The present invention belongs to the technical field of the preparation of powder metallurgy, in particular to provide a method for preparing a copper tungsten light purple modified catalyst.

Background technique

With the increasingly serious energy and environmental problems, including organic pollution of water resources is particularly prominent, not only affects human health, but also need to consume large amounts of energy to eliminate such contaminants. Therefore, the development of new materials photocatalytic technology to solve the pollution has become a new study based on the direction. From Japan, Honda and Fujishima found that by single-crystal T12 photocatalytic electrode can begin to decompose water, research traitor traitor who constantly research new efficient catalyst material. WO 3, T12, ZnO, Fe2O3 and a series of semiconductor oxide because the band gap energy of the sun is not a good match, so when needed as a catalyst modified. Surface modification method photosensitizing doped metal ions, noble metal deposition, compound semiconductors, and composite insulators, quantization and a series of methods. The development of a photocatalyst, it should have a low production cost, high secondary use, stable performance for efficient use of solar energy, to improve the catalytic efficiency of the photocatalyst. Among catalyst materials, purple tungsten has a special surface structure, chemical activity, large concentrations of oxygen defect structure, and has a higher degree of absorption in ultraviolet light, while research shows that copper as a traitor doping element can be a good It increased catalyst in the visible range of the response. Therefore, the

choice of purple tungsten doped with copper as the photocatalytic material has a good prospect.

Currently, the photocatalyst material prepared by transition metal-doped sol-gel method mainly, hydrothermal method, precipitation method, microemulsion method, gas method. Chinese Patent No. CN101898139 A discloses a method for preparing titania-doped tungsten oxide method. By titanium dioxide dispersed in the aqueous ammonia solution in acid, after drying to obtain a mixed powder, then the powder is dispersed in a concentration of sulfate solution and then dried. The method is simple and convenient, but because it is the use of the second solid-liquid mixing, will result in a uniform mixing between the powder is not enough, after drying the dispersion is insufficient. China Patent CN102985178 A discloses a dispersion method of copper ion-modified tungsten oxide photocatalyst. By copper ions in solution and the tungsten oxide particles mechanical polishing, and then the dispersion with oxygen or ozone exposure. The method is simple, low cost and high efficiency. But the same because it is a solid-liquid mixing, resulting in insufficient mixing uniformity. In short, the preparation of doped tungsten oxide photocatalyst Some of these methods long preparation time, some method has particle size and distribution of non-uniform pore structure, high cost and other issues, some preparation of the catalyst. Therefore, the need to develop new low-cost, high-efficiency light catalyst.

SUMMARY OF THE INVENTION

The object of the present invention is to provide a low cost, tungsten light purple new method is simple and efficient method for catalyst preparation of copper-doped.

The present invention is directed to the use of tungstate (ammonium paratungstate) and copper nitrate as raw materials, with a short process, low cost, and to prepare a copper-doped tungsten light purple powder catalyst particle size of nanometer level, and mixing uniform, high catalytic efficiency.

The process steps of the present invention are as follows:
1, raw materials: the raw materials used tungsten source ((NH4) 6H2W12O40), copper source (Cu (NO3) 2), ammonium nitrate (NH4NO3), adjuvants.
2, solution preparation: raw dubbed the selected solution in accordance with a certain proportion, mix well.
3, precursor preparation: heating the aqueous solution obtained in step 2, with the evaporation of water, the solution was concentrated in the event bubbling and emit a lot of gas, occurred within tens of seconds vigorous oxidation-reduction reaction, to generate the precursor powder.

The molar ratio of [0010] wherein the step of copper nitrate and crane source 2 is 0.3~1, the molar ratio of nitric acid and crane source of money for 12~36; molar ratio of adjuvant and tungsten source for 5 to 15.

Advantages of the present invention are:

1, The direct use of tungstate (ammonium paratungstate) and copper nitrate as raw materials, short process, low cost, simple process, quick, suitable for large-scale production.

2, You can control the type and proportion of raw materials, control of the reaction process Precursors control powder morphology and crystalline form.

3, Mixing the raw material by liquid, can achieve uniform mixing of the reactants at the atomic level, to facilitate a uniform distribution of pore structure obtained.

4. The process of the invention to produce large amounts of gas, effective control of the contact reaction process reactant and oxygen to generate Violet Tungsten plays an important role.

5, since the present invention, a low copper content, to ensure that the copper ions into the crystal lattice of tungsten purple, rather than forming a mixture of tungsten oxide, copper and purple. Use of copper ions doped tungsten purple introducing defects in the crystal lattice position, thus affecting the recombination of electrons and holes. In appropriate concentrations to achieve optimal catalytic effect.

Example 1

The reaction weighed stoichiometric ratio of copper nitrate (Cu (NO3) 2) 0.0lmol, ammonium metatungstate ((NH4) 6H2W1204 (l) 0.0lmol, ammonium nitrate (NH4NO3) 0.24mol, urea (CO (NH2) 2) 0.1mol0 raw material powder is dissolved in an appropriate amount of deionized water with stirring until completely dissolved, heating thermostat 200 ° C, the solution was evaporated to dryness after the redox reaction occurs violently purple copper-doped tungsten powder within tens of seconds. The resulting powder nano-needles, diameter 30~150nm, length I~3 μ m, at a concentration of lg / L of the catalyst 20ppm organic pollutants (methylene blue) End degradation need 40min.

Example 2

The reaction weighed stoichiometric ratio of copper nitrate (Cu (NO3) 2) 0.0lmol, ammonium metatungstate ((NH4) 6H2W1204 (l) 0.0lmol, ammonium nitrate (NH4NO3) 0.24moI, urea (CO (NH2) 2) 0.15mol. The raw material powder was dissolved in an appropriate amount of deionized water with stirring until completely dissolved, heating thermostat 200 ° C, the solution was evaporated to dryness after the redox reaction occurs violently purple copper-doped tungsten powder within tens of seconds. The resulting nano powder needle diameter 50~150nm, length I~3 μ m, at a concentration of lg / L of the catalyst 20ppm organic pollutants (methylene blue) complete degradation need 35min.

Example 3

The reaction weighed stoichiometric ratio of copper nitrate (Cu (NO3) 2) 0.0lmol, ammonium metatungstate ((NH4) 6H2W1204 (l) 0.0lmol, ammonium nitrate (NH4NO3) 0.36mol, urea (CO (NH2) 2) 0.1mol0 After the mixture was stirred until completely dissolved material powder was dissolved in an appropriate amount of deionized water, heating thermostat 200 ° C, the solution was evaporated to dryness after the occurrence of the vigorous oxidation-reduction reaction within several tens of seconds to generate purple copper doped tungsten powder. The

resulting nano powder needle diameter 60~150nm, length I~2 μ m, at a concentration of lg / L of the catalyst 20ppm organic pollutants (methylene blue) complete degradation need 50min.

Example 4

The reaction weighed stoichiometric ratio of copper nitrate (Cu (NO3) 2) 0.0lmol, ammonium metatungstate ((NH4) 6H2W1204 (l) 0.0lmol, ammonium nitrate (NH4NO3) 0.24mol, citric acid (C6H8O7) 0.08 moI. After the mixture was stirred until completely dissolved material powder was dissolved in an appropriate amount of deionized water, heating thermostat 200 ° C, a redox reaction occurs violently solution was evaporated to dryness in tens of seconds to generate purple copper doped tungsten powder. The resulting powder Nano needle diameter 30~150nm, length I~3 μ m, at a concentration of lg / L of the catalyst 20ppm organic pollutants (methylene blue) End degradation need 50min.

Example 5

The reaction weighed stoichiometric ratio of copper nitrate (Cu (NO3) 2) 0.0lmol, ammonium metatungstate ((NH4) 6H2W1204 (l) 0.0lmol, ammonium nitrate (NH4NO3) 0.18mol, citric acid (C6H8O7) 0.15 mol. After the mixture was stirred until completely dissolved material powder was dissolved in an appropriate amount of deionized water, heating thermostat 200 ° C, the solution was evaporated to dryness after the occurrence of the vigorous oxidation-reduction reaction in a matter of seconds to generate purple copper doped tungsten powder. The resulting powder is nano needle diameter 80~200nm, length I~2 μ m, at a concentration of lg / L of the catalyst 20ppm organic pollutants (methylene blue) complete degradation need 60min.