METHOD FOR PREPARING A NANO MGO ACTIVATED CARBON FOR ADSORBING HEAVY METALS

The present invention relates to the field of water purification, and discloses a method for preparing a nano MgO activated carbon for adsorbing heavy metals. Specifically, the method comprises the following steps: a. treating activated carbon with dilute nitric acid, then washing in water and anhydrous solvent, and drying at 60 to 80°C, to obtain treated activated carbon; b. providing an aqueous Mg(NO₃)₂ solution with deionized water as a solvent; c. adding the treated activated carbon obtained from step a to the aqueous Mg(NO₃)₂ solution obtained from step b, and stirring to mix evenly; d. preparing an aqueous urea solution, then dripping the aqueous urea solution into the solution processed in step c, and stirring to obtain a liquid mixture; e. drying the activated carbon processed in step d at 60 to 80°C; and f. calcining the activated carbon processed in step e to obtain nano MgO activated carbon for adsorbing heavy metals. In the present invention, flower-like nano MgO is loaded on the surface or in the voids of activated carbon by a chemical deposition method; this can not only preserve the intrinsic characteristics of a nano material, but also enhance the stability thereof, and the heavy metals lead and cadmium in drinking water can be adsorbed efficiently.
METHOD FOR PREPARING A NANO MGO ACTIVATED CARBON FOR ADSORBING HEAVY METALS

Field of the invention

The present invention relates to the field of water purification, in particular to a method for preparing a nano MgO activated carbon capable of adsorbing heavy metals in water.

Background of the invention

Rivers, lakes and reservoirs are the main forms of surface water used as drinking water sources. Monitoring and analysis of these types of bodies of water has shown that these forms of surface water have already been contaminated with heavy metals such as lead and cadmium. Heavy metals such as lead and cadmium are easily concentrated in organisms through the food chain, and constitute a serious threat to the health of organisms and the human body. Pollution of the water from water sources not only causes considerable harm to the health of organisms and the human body, but also has a significant effect on the water quality and conventional water purification processes of water supply plants. As people's quality of life continuously improves and progress is made in means of detection and analysis, people's demands on drinking water quality will become more stringent, and corresponding water quality standards for water supply are also continuously rising. Thus, the question of how to effectively remove heavy metals such as lead and cadmium from drinking water has become an environmental problem that is currently in urgent need of a solution.

As an alkaline agent, more correctly an alkaline agent with very strong buffering properties and efficient chemical adsorbency, MgO is receiving more and more attention. In recent years, there has been much research and development in connection with the applications of magnesium agents in different fields, and applications in the environmental field have topped the list. They show excellent results in the treatment of wastewater from printing and dyeing as well as acidic wastewater for example, and especially in the removal of heavy metal ions, so are revered as environmentally friendly chemicals.
Summary of the invention

First aspect of the present invention provides a method for preparing a nano MgO activated carbon for adsorbing heavy metals, the method comprising steps of:

a. treating activated carbon with dilute nitric acid, then washing in water and anhydrous solvent, and drying at 60 to 80°C, to obtain treated activated carbon;

b. providing an aqueous Mg(NC>3)2 solution with deionized water as a solvent;

c. adding the treated activated carbon obtained from step a to the aqueous Mg(NC>3)2 solution obtained from step b, and stirring to mix evenly;

d. preparing an aqueous urea solution, then dripping the aqueous urea solution into the solution processed in step c, and stirring to obtain a liquid mixture;

e. drying the activated carbon processed in step d at 60 to 80°C; and

f. calcining the activated carbon processed in step e to obtain nano MgO activated carbon for adsorbing heavy metals.

Second aspect of the present invention provides a nano MgO activated carbon obtained by the method according to first aspect.

Third aspect of the present invention provides a filter core, comprising a filter core body, and a nano MgO activated carbon, wherein the nano a-Fe203 modified activated carbon is prepared by the method according to first aspect and is packed in the filter core body.

Fourth aspect of the present invention provides use of nano MgO activated carbon obtained by any method according to first aspect for adsorbing heavy metals.

Fifth aspect of the present invention provides use of nano MgO activated carbon obtained by any method according to first aspect for removal of heavy metals from water.

Brief Description of drawings

Fig. 1 is a curve of a first determination method.
Fig. 2 is a curve of a second determination method.
Fig. 3 is a curve of a third determination method.
Fig. 4 is a curve of a fourth determination method.

**Detailed description of the invention**

In response to the problem of removing heavy metals such as lead and cadmium from drinking water in the prior art, the present invention provides a method for preparing a nano MgO activated carbon for adsorbing heavy metals.

The term nano MgO means nano sized MgO, which is magnesium oxide Nanomaterials with diameters of <100 nm. To solve the abovementioned technical problem, the present invention employs the following technical solution:

The present invention provides a method for preparing a nano MgO activated carbon for adsorbing heavy metals, the method comprising steps of:

a. treating activated carbon with dilute nitric acid, then washing in water and anhydrous solvent, and drying at 60 to 80°C, to obtain treated activated carbon;
b. providing an aqueous Mg(NO₃)₂ solution with deionized water as a solvent;
c. adding the treated activated carbon obtained from step a to the aqueous Mg(NO₃)₂ solution obtained from step b, and stirring to mix evenly;
d. preparing an aqueous urea solution, then dripping the aqueous urea solution into the solution processed in step c, and stirring to obtain a liquid mixture;
e. drying the activated carbon processed in step d at 60 to 80°C; and
f. calcining the activated carbon processed in step e to obtain nano MgO activated carbon for adsorbing heavy metals.

It is preferred that the present invention provides a method of the present invention, wherein the anhydrous solvent is anhydrous ethanol.

It is preferred that the molar ratio of Mg(NO₃)₂ to urea in the liquid mixture is 1:2.
It is preferred that volume ratio of anhydrous solvent to water in step a ranges from 1:3 to 1:1, and the washing time in each case ranges from 0.5 to 2 hours.

It is preferred that treatment of activated carbon in dilute nitric acid comprises immersing the activated carbon in dilute nitric acid for a period of 0.5 to 2 hours.

It is preferred that in step c, stirring is performed for 2.5 to 5.5 hours at a temperature ranging from 45 to 50°C.

It is preferred that the stirring in step c is performed when the temperature has been increased to 45 to 50°C.

It is preferred that in step d, after dripping the aqueous urea solution into the solution processed in step c, the pH is adjusted in the range of 8.0 to 10.0.

It is preferred that in step d, NaOH is used to adjust the pH.

It is preferred that the calcining process in step f comprises: increasing temperature to 350 to 400°C at a rate of 3 to 5°C/min, and maintaining the temperature for 1.5 to 4.5 hours.

The present invention provides a nano MgO activated carbon obtained by any method of the present invention.

The present invention also provides a filter core, comprising a filter core body, and a nano a-Fe203 modified activated carbon, wherein the nano a-Fe203 modified activated carbon is prepared by the method according to the present invention and is packed in the filter core body.

The present invention also provides use of nano MgO activated carbon obtained by method according to the present invention for adsorbing heavy metals.

The present invention also provides use of nano MgO activated carbon obtained by any method according to first aspect for removal of heavy metals from water.
It is preferred that the method for preparing a nano MgO activated carbon for adsorbing heavy metals comprises steps of:

a. immersing activated carbon in dilute nitric acid, then washing alternately in water and anhydrous ethanol, to remove substances adhering to the surface; sun-drying, air-drying, or oven-drying at 60 - 80°C, to remove surplus water from the surface or voids of the activated carbon, in preparation for use;
b. using Mg(NC>3)2-6H2O as a precursor for producing MgO, making up a colourless transparent aqueous Mg(NC>3)2 solution with deionized water as a solvent;
c. adding the activated carbon processed in process step a to the aqueous Mg(NC>3)2 solution processed in step b, and stirring to mix evenly;
d. weighing out a certain amount of urea as an alkaline liquid for producing MgO, making up an aqueous urea solution therefrom, then dripping the formulated aqueous urea solution into the solution processed in step c, and stirring;
e. sun-drying, air-drying, or oven-drying at 60 - 80°C the activated carbon processed in step d;
f. putting the activated carbon processed in step e into a calcining furnace and calcining to obtain nano MgO activated carbon for adsorbing heavy metals; calcining can cause the MgO to assume a special crystal form and thereby have a strong capacity for adsorbing heavy metals.

Preferably, the volume ratio of anhydrous ethanol to water in step a is 1:3 to 1:1, and the washing time in each case is 1 h.

Preferably, the concentration of dilute nitric acid in step a is 0.1 M, wherein 100 g of activated carbon is washed in 1 L of this dilute nitric acid, the immersion time being 0.5 to 2 h.

Preferably, heating is controlled by a water bath in step c, such that stirring is performed for 2.5 to 5.5 h at a temperature of 45 to 50°C, because the aqueous Mg(NO3)2 solution can be fully adsorbed on the surface or in the voids of the activated carbon at this temperature.
Preferably, in step d, after dripping the formulated aqueous urea solution into the solution processed in step c, the pH is adjusted to 8.0 to 10.0 using 0.01 M NaOH; within this pH range, the Mg(NC>3)2 can be completely converted to magnesium hydroxide, and this is a prerequisite for the formation of MgO.

Preferably, a calcining process in step f is as follows: increasing the temperature to 350 to 400°C at a rate of 3 to 5°C/min, and maintaining this temperature for 1.5 to 4.5 h.

Due to the adoption of the technical solution described above, the present invention has the following significant technical effects:

In the present invention, flower-like nano MgO is loaded on the surface or in the voids of activated carbon by a chemical deposition method; this can not only preserve the intrinsic characteristics of a nano material, but also enhance the stability thereof, and the heavy metals lead and cadmium in drinking water can be adsorbed efficiently.

The present invention is described in further detail below in conjunction with the accompanying drawings and embodiments.

It is preferred that the method for preparing a nano MgO activated carbon for adsorbing heavy metals, comprises the following steps:

a. immersing 10 g of activated carbon in 100 mL of dilute nitric acid with a concentration of 0.1 M for 0.5 h, then washing alternately in water and anhydrous ethanol, wherein the volume ratio of anhydrous ethanol to water is 1 : 3, and the washing time in each case is 0.5 h, to remove substances adhering to the surface, and oven-drying at 60°C, in preparation for use;

b. using Mg(NO3)2-6H2O as a precursor, making up a 0.1 M aqueous Mg(NO3)2 solution with deionized water as a solvent;

c. adding the activated carbon processed in process step a to the aqueous Mg(NO3)2 solution processed in step b, and stirring for 2.5 h in a 45°C water bath to mix evenly;

d. step d: weighing out 1.2 g of urea, making up an aqueous urea solution with a concentration of 0.2 M, then dripping the formulated aqueous urea solution into
the solution processed in step c, stirring and using 0.01 M NaOH to adjust the pH to 8.0 - 10.0;
e. step e: sun-drying the activated carbon processed in step d;
f. putting the activated carbon processed in step e into a calcining furnace and calcining, increasing the temperature to 350°C at a rate of 3°C/min, and maintaining this temperature for 1.5 h, to obtain nano MgO activated carbon for adsorbing heavy metals.

The present invention provides use of the nano MgO activated carbon for adsorbing heavy metals in the removal of heavy metals from drinking water: original water containing the heavy metal lead or cadmium is made up using deionized water, and after having its flow speed adjusted by an electromagnetic flow meter, passes through nano flower-like MgO material before coming out; the original water and the water coming out are respectively sampled to measure the change in heavy metal concentration, wherein the specific determination methods are divided into the following types:

First type: Original waters with initial heavy metal lead concentrations of 5 mg/L, 10 mg/L and 15 mg/L are respectively made up, and after passing through nano MgO activated carbon, come out; the incoming water flow speed is 100 mL/min, and water is sampled at preset time points to measure the change in lead concentration thereof. See Fig. 1 for specific results.

Second type: Original waters with initial heavy metal cadmium concentrations of 5 mg/L, 10 mg/L and 15 mg/L are respectively made up, and after passing through nano MgO activated carbon, come out; the incoming water flow speed is 100 mL/min, and water is sampled at preset time points to measure the change in cadmium concentration thereof. See Fig. 2 for specific results.

Third type: Original water with an initial heavy metal lead concentration of 5 mg/L is made up, and after passing through nano MgO activated carbon, comes out; the incoming water flow speed is set to 1000 mL/min and 2000 mL/min respectively, and water is sampled at preset time points to measure the change in lead concentration thereof. See Fig. 3 for specific results.
Fourth type: Original water with an initial heavy metal cadmium concentration of 5 mg/L is made up, and after passing through nano MgO activated carbon, comes out; the incoming water flow speed is set to 1000 mL/min and 2000 mL/min respectively, and water is sampled at preset time points to measure the change in cadmium concentration thereof. See Fig. 4 for specific results.

Considering all of the above, it is clear that the ability of the nano MgO activated carbon to remove the heavy metals lead and cadmium is unrelated to flow speed, but in actual applications, flow speed is often an important factor restricting material applications. The ability of the nano MgO activated carbon to adsorb the heavy metals chromium and arsenic will not change significantly with a change in flow speed, therefore there is no need to take flow speed into account when designing a water purification apparatus; this material has very strong prospects for actual application. Furthermore, the preparation and application of this material involve simple operations, and it is very easy to industrialize, so has broad application prospects in the field of the removal of heavy metals from drinking water.

The present invention also provides a method for preparing a nano MgO activated carbon for adsorbing heavy metals, comprising the following steps:

a. immersing 10 g of activated carbon in 100 mL of dilute nitric acid with a concentration of 0.1 M for 2 h, then washing alternately in water and anhydrous ethanol, wherein the volume ratio of anhydrous ethanol to water is 1 : 1, and the washing time in each case is 2 h, to remove substances adhering to the surface, and sun-drying, in preparation for use;
b. using Mg(NO₃)₂·6H₂O as a precursor, making up a 0.1 M aqueous Mg(NO₃)₂ solution with deionized water as a solvent;
c. adding the activated carbon processed in process step a to the aqueous Mg(NO₃)₂ solution processed in step b, and stirring for 5.5 h in a 50°C water bath to mix evenly;
d. weighing out 1.2 g of urea, making up an aqueous urea solution with a concentration of 0.2 M, then dripping the formulated aqueous urea solution into the solution processed in step c, stirring and using 0.01 M NaOH to adjust the pH to 8.0 - 10.0;
e. oven-drying at 80°C the activated carbon processed in step d;

f. putting the activated carbon processed in step e into a calcining furnace and calcining, increasing the temperature to 400°C at a rate of 5°C/min, and maintaining this temperature for 4.5 h, to obtain nano MgO activated carbon for adsorbing heavy metals.

In summary, the embodiments above are merely preferred embodiments of the present invention. All equivalent changes and modifications made within the patent application scope of the present invention shall be included in the scope of the present invention patent.
Claims

1. A method for preparing a nano MgO activated carbon for adsorbing heavy metals, the method comprising the following steps:
   a. treating activated carbon with dilute nitric acid, then washing in water and anhydrous solvent, and drying at 60 to 80°C, to obtain treated activated carbon;
   b. providing an aqueous Mg(NC>3)2 solution with deionized water as a solvent;
   c. adding the treated activated carbon obtained from step a to the aqueous Mg(NC>3)2 solution obtained from step b, and stirring to mix evenly;
   d. preparing an aqueous urea solution, then dripping the aqueous urea solution into the solution processed in step c, and stirring to obtain a liquid mixture;
   e. drying the activated carbon processed in step d at 60 to 80°C; and
   f. calcining the activated carbon processed in step e to obtain nano MgO activated carbon for adsorbing heavy metals.

2. A method according to claim 1, wherein the anhydrous solvent is anhydrous ethanol.

3. A method according to claims 1 or 2, wherein the molar ratio of Mg(NC>3)2 to urea in the liquid mixture is 1 : 2.

4. A method according to anyone of the preceding claims 1 to 3, wherein volume ratio of anhydrous solvent to water in step a ranges from 1:3 to 1:1, and the washing time in each case ranges from 0.5 to 2 hours.

5. A method according to anyone of the preceding claims from 1 to 4, wherein the treatment of activated carbon in dilute nitric acid comprises immersing the activated carbon in dilute nitric acid for a period of 0.5 to 2 hours.

6. A method according to anyone of the preceding claims from 1 to 5, wherein, in step c, stirring is performed for 2.5 to 5.5 hours at a temperature ranging from 45 to 50°C.

7. A method according to anyone of the preceding claims from 1 to 6, wherein the stirring in step c is performed when the temperature has been increased to 45 to 50°C.
8. A method according to anyone of the preceding claims from 1 to 7, wherein, in step d, after dripping the aqueous urea solution into the solution processed in step c, the pH is adjusted in the range of 8.0 to 10.0.

9. A method according to anyone of the preceding claims from 1 to 7, wherein in step d, NaOH is used to adjust the pH.

10. A method according to anyone of the preceding claims from 1 to 9, wherein the calcining process in step f comprises: increasing temperature to 350 to 400°C at a rate of 3 to 5°C/min, and maintaining the temperature for 1.5 to 4.5 hours.

11. A nano MgO activated carbon obtained by any method according to anyone of the preceding claims from 1 to 10.

12. A filter core, comprising a filter core body, and a nano MgO activated carbon, wherein the nano a-Fe2O3 modified activated carbon is prepared by the method according to anyone of the preceding claims 1 to 10 and is packed in the filter core body.

13. Use of nano MgO activated carbon obtained by any method according to anyone of the preceding claims from 1 to 10 for adsorbing heavy metals.

14. Use of nano MgO activated carbon obtained by any method according to anyone of the preceding claims from 1 to 10 for removal of heavy metals from water.
### B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols):

- B01J
- C02F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched.

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used):

- **EPO-Internal, WPI Data**

### C. DOCUMENTS CONSIDERED TO BE RELEVANT

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<td>EP 0 311 244 AI (KUREHA CHEMICAL IND CO LTD [JP]) 12 April 1 1989 (1989-04-12) claims 1-6 page 3, line 41 - line 43 examples 1,2,7 page 4, line 12 - line 17 -----</td>
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**X** Further documents are listed in the continuation of Box C. **X** See patent family annex.

- * Special categories of cited documents:
  - "A" document defining the general state of the art which is not considered to be of particular relevance
  - "E" earlier application or patent but published on or after the international filing date
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**Date of the actual completion of the international search:** 31 October 2017

**Date of mailing of the international search report:** 08/11/2017

**Authorized officer:** Kal uza, N i col eta
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<td>IPSITA HAZRA CHOWDHURY ET AL: &quot;Effect of anion type on the synthesis of mesoporous nanostructured MgO, and its excellent adsorption capacity for the removal of toxic heavy metal ions from water&quot; , RSC ADV., vol. 6, no. 8, 1 January 2016 (2016-01-01) , pages 6038-6047 , XP055420624, DOI : 10.1039/C5RA16837F abstract 2.2 Synthesis of MgO nanostructures ; page 6038, left-hand column 3.2 Heavy metal ion adsorption study; page 6043, right-hand column - page 6047</td>
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