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National Food Safety Standard on Vegetable Activated Carbon

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Report Highlights:

On November 18, 2011, China notified the WTO of National Food Safety Standard: Food Additive Vegetable Activated Carbon (Wooden Activated Carbon) as SPS/N/CHN/495. This standard applies to food additive vegetable activated carbon (wooden activated carbon) made from plant materials such as sawdust, coconut shells, and nutshells through physical or chemical method. The date for submission of final comments to China is January 17, 2012. The proposed date of entry is to be determined. Comments can be sent to China's SPS Enquiry Point at sps@aqsiq.gov.cn. This report is an INFORMAL translation of this document.

General Information:

BEGIN TRANSLATION

GB National Food Safety Standard
GB XXXX－XXXX

National Food Safety Standard

Food Additive Plant-based Activated Carbon (Wood Activated Carbon) (Draft for Soliciting Opinions)

Date of Issue: XXXX-XX-XX

Date of Effectiveness: XXXX-XX-XX

Issued by the Ministry of Health of the People's Republic of China

National Food Safety Standard
Food Additive Plant-based Activated Carbon
(Wood Activated Carbon)

1. Scope

This standard applies to the food additive, plant-based activated Carbon (Wood Activated Carbon), prepared from the plant-based materials such as the saw dust, the coconut shell and nut shell through physical or chemical methods.

2. Technical requirements

Organoleptic requirements: should conform to the requirements in table 1.

Table 1: Organoleptic Requirements

Item	Requirement	Method of inspection
Color	Black	Apply adequate amount of sample into a 50 mL beaker and observe the color and texture status under natural lighting
Texture	Particles or powders	

The physical and chemical indexes: shall conform to the requirements in Table 2.

Table 2. The physical and chemical indexes

Item	Index	Method of inspection

Iodine Adsorption Value (in dry basis) (mg/g) ≥	400	GB/T 12496.8
Loss on drying w/% ≤ ≤	To be negotiated	A.4 in Appendix A
Sulfated ash content (in dry basis) w/% ≤	5.0	A.5 in Appendix A
Cyanide test (in dry basis)	Test passed	GB/T 12496.14
Senior aromatic hydrocarbon test (in dry basis)	Test passed	A.6 in Appendix A
Hydrotrope (in dry basis) w/% ≤	4.0	A.7 in Appendix A
Arsenic (As) (in dry basis) / (mg/kg) ≤	3	A.8 in Appendix A
Plumbum (Pb) (in dry basis) / (mg/kg) ≤	10	A.9 in Appendix A
Note: Sulfated ash content of the activated carbon made by H3P04 method shall not be more than 7.0%		

Appendix A

Method of inspection

A.1 Cautions

Some of the reagents as stipulated by the testing method hereof are toxic or corrosive, be careful in operation! In case of contact with skin, immediately flush with water. In serious cases, must be treated immediately. When using inflammables, open flame is strictly prohibited for heating.

A.2 General Provision

Unless otherwise specified, all the reagents and water used hereof shall refer to the reagents identified as A.R and the Class III water as defined in GB/T6682-2008. When no other requirement is noted, all the standard titration solution, the standard solution, preparation and products for determination of impurities that will be used by the testing method shall be prepared as prescribed in GB/T 601, GB/T 602 and GB/T 603.

A.3 Identification

A.3.1 Reagents and materials

A.3.1.1 Hydrochloric acid solution: the mass fraction is 5%.

A.3.1.2 Iodine solution;

Dissolve 14 g iodine and 36 g potassium iodide in 100 mL water, add in three drops of hydrochloric acid, and dilute it with water to 1,000 mL, shaking evenly.

A.3.2 Identification steps

Weigh 3.00 g sample, accurate to 0.01 g, put it into an iodine number flask with 10 mL hydrochloric acid solution, boil it and maintain for 30 s, cooled to room temperature, add in 100 mL Iodine solution, and put on the bottle cork, shaking heavily for 30 s. It is filtered through moderate-speed qualitative filter paper, and abandon 20 Ml primary filtrate; transfer the filtrate into a 50 mL colorimetric cylinder to the scale, compare it with the reference solution; the color shall not be darker than that of the reference solution.

Reference solution： Transfer 10 mL Iodine solution by transfer pipet into a 50 mL colorimetric cylinder and dilute it with water to the scale, shaking evenly.

A.4 Determination of Loss on drying

A.4.1 Instruments and devices

Weighing bottle: Φ50 mm×30 mm

A.4.2 Analysis steps

Weigh 1 g～2 g sample, accurate to 0.0002 g by the weighing bottle that has been dried to constant mass at 120 ℃±2 ℃, which shall be dried for 4 hours 120 ℃±2 ℃. Put it into a desiccator, cooling to room temperature and then weigh it.

A.4.3 Result calculation

The Loss on drying is calculated by the mass fraction of w1, and the numerical value is indicated by %, which is calculated according to formula (A.1):

$$w1 = \frac{m_1 - m_2}{m} \times 100\% \dots\dots (A.1)$$

Where: m_1 = the numerical value of the mass of the sample and the weighing bottle before drying, in the unit of gram (g);

m_2 = the numerical value of the mass of the weighing bottle and the sample before drying, in the unit of gram (g);

m= the numerical value of the mass of the sample, in the unit of gram (g).

The arithmetic mean of the result of two measurements in the same condition is taken as the determined result, where the absolute difference between the results of two measurements in the same condition shall not be more than 2%.

A.5. Determination of Sulfated ash content

A.5.1 Reagents and materials

A.5.1.1 Ammonium carbonate.

A.5.1.2 Sulphuric acid.

A.5.1.3 Sulphuric acid solution: 1+19.

A.5.2 Instruments and devices

A.5.2.1 Porcelain crucible: 30 mL.

A.5.2.2 High temperature furnace: enable to control the temperature at 800℃±25℃.

A.5.3 Analysis steps

Weigh 0.5 g sample, accurate to 0.0002 g, put it into the Porcelain crucible that has been dried to constant mass at 800℃±25℃ in advance, add in sufficient Sulphuric acid solution to infiltrate all the sample, which is placed onto a hot plate to be heated slowly until the sample is dried and carbonized completely; further heat until all the sample is volatilized and almost all the carbon is oxidized, cooled to room temperature. Add in 0.1 mL sulphuric acid to infiltrate the residue and further heat it until the residues and superfluous sulphuric acid are vaporized according to abovementioned method; a few grains of Ammonium carbonate can be added into the residues of the sulphuric acid so as to promote the volatilization of sulphuric acid; further igniting at 800℃±25℃ to constant mass.

A.5.4 Result calculation

The **Sulfated ash content** is calculated by the mass fraction of w2 and the numerical value is indicated by %, which is calculated according to formula (A.2):

$$W2 = \frac{m1-m2}{m \times \frac{(100-w1)}{100}} \times 100\% \text{...} \quad (A.2)$$

Where:

$m1$ = the numerical value of the mass of the residues and the crucible after igniting, in the unit of gram (g);

$m2$ = the numerical value of the mass of the crucible, in the unit of gram (g);

m = the numerical value of the mass of the sample, in the unit of gram (g);

w1=the mass fraction of the loss on drying determined according to A.4, the numerical value is indicated by %.

The arithmetic mean of the result of two measurements in the same condition is taken as the determined result, where the absolute difference between the results of two measurements in the same condition shall not be more than 0.50%.

A.6 Senior aromatic hydrocarbon test

A.6.1 Reagents and materials

A.6.1.1 Cyclohexane

A.6.1.2 The standard stock solution I of quinine sulfate: 1 mL solution contains 1 mg quinine sulfate $[(C_{20}H_{24}N_2O_2)_2\cdot H_2SO_4]$;

Weigh 1.048 g quinine sulfate $[(C_{20}H_{24}N_2O_2)_2\cdot H_2SO_4]$, put it into a 100 mL volumetric flask, dissolve it into sulphuric acid solution (3+1,000), use sulphuric acid solution (3+1,000) to dilute it to the scale, shaking evenly.

A.6.1.3 The standard stock solution II of quinine sulfate: 1 mL solution contains 0.01 mg quinine sulfate $[(C_{20}H_{24}N_2O_2)_2\cdot H_2SO_4]$;

Transfer 1 mL standard stock solution I of quinine sulfate by a transfer pipet, put it into a 100 mL volumetric flask, use sulphuric acid solution (3+1,000) to dilute it to the scale, shaking evenly.

A.6.1.4 The standard using solution of quinine sulfate: 1 mL solution contains 1 μg quinine sulfate $[(C_{20}H_{24}N_2O_2)_2\cdot H_2SO_4]$;

Transfer 1 mL standard stock solution I of quinine sulfate by a transfer pipet, put it into a 100 mL volumetric flask, use sulphuric acid solution (3+1,000) to dilute it to the scale, shaking evenly. This solution shall be prepared instantly and used instantly.

A.6.2 Instruments and devices

A.6.2.1 Soxhlet extractor

A.6.2.2 Colorimetric cylinder: 10 mL.

A.6.3 Analysis steps

Weigh 1.00 g±0.01 g dried sample that has been crushed to 71 μm, use clean filter paper to wrap it tightly, put it into the Soxhlet extractor, and extract it continuously by using 12.0 mL cyclohexane for two hours. When cooled, put the extracting solution into a colorimetric cylinder.

The standard solution is that 10 mL of the standard using solution of quinine sulfate is put into a colorimetric cylinder.

When observing under the UV Lamp, if the color or the fluorescence displayed from the sample solution does not surpass that of the standard solution, it shall be deemed as passing the test.

A.7 Determination of Hydrotrope content

A.7.1 Instruments and devices

A.7.1.1 Reflux condenser

A.7.1.2 Electrothermal constant temperature dry box: enable to control the temperature 100℃±2℃.

A.7.2 Analysis steps

Pick up 5 g of the dried sample that has been crushed to 71 μm, accurate to 0.01 g. put it into a 250 mL dried beaker with Reflux condenser and Bunsen valve. Add in 100.0 mL water and several glass balls, and heat to reflux for 1 hour. Cooling slightly, it is dryly filtered through moderate-speed qualitative filter paper and abandon 10 mL of the initial filtrate. Cool the rest filtrate to room temperature. Transfer 25 mL filtrate by a transfer pipet, put it into an evaporating dish that has been dried to constant mass at 100 ℃ ±2 ℃. Put the evaporating dish onto a water bath for evaporation until the filtrate is almost dried, during which the solution shall not be heated to boiling. It is dried to constant mass at 100 ℃ ±2 ℃. The rest filtrate is retained for determination of arsenic.

A.7.3 Result calculation

The **Hydrotrope content** is calculated by the mass fraction of w3 and the numerical value is indicated by %, which is calculated according to formula (A.3):

$$W2 = \frac{m1-m2}{m \text{ x } (25/100)} \text{ x}100\% \ldots\ldots\ldots\ldots \text{(A.3)}$$

Where:

$m1$ = the numerical value of the mass of the residues and the evaporating dish after drying, in the unit of gram (g);

$m2$ = the numerical value of the mass of the evaporating dish, in the unit of gram (g);

m = the numerical value of the mass of the sample, in the unit of gram (g);

A.8 Determination of arsenic content

Pick up 20.00 mL filtrate retained during the determination of Hydrotrope content, put it into a arsenic determination apparatus as the testing solution.

Pick up 3.00 mL arsenic standard solution (1 mL arsenic contains 0.001 mg As) as the standard solution of arsenic limit. The operation shall be conducted according to Chapter XI, the arsenic stain method, in GB/T 5009.76－2003.

A.9 Determination of plumbum content

A.9.1 Summary of method

Use the water containing thin nitric acid to extract the plumbum ion, where the determination can be conducted through an atomic absorption spectrophotometer, by way of air-acetylene flame and by using the work curve method.

A.9.2 Reagents and materials

A.9.2.1 Nitric acid

A.9.2.2 Nitric acid solution: 1+5.

A.9.2.3 Plumbum standard solution: 1 mL solution contains 0.10 mg of plumbum (Pb).

A.9.2.4 Class II water: in line with the provisions of GB/T 6682—2008.

A.9.3 Instruments and devices

Atomic absorption spectrophotometer: provided with Pb hollow cathode lamp.

A.9.4 Determination steps

A.9.4.1 Instruments cleaning

All the glass ware shall be dipped in Nitric acid solution (1+5) overnight, then rinsed by tap water repeatedly and finally rinsed up by Class II water.

A.9.4.2 Drafting the work curve

Use the transfer pipet to pick up 0 mL, 0.50 mL, 1.00 mL and 1.50 mL of plumbum standard solution, respectively put them into four 50-mL volumetric flasks, add in 1 mL nitric acid and dilute them with Class II water to the scale, shaking evenly. On the atomic absorption spectrophotometer, choose air-acetylene flame, use the Class II water to adjust to zero at the wavelength of 283.3 nm; choosing the best working condition of the instrument, determine the absorbency of the abovementioned solutions.

Reduce the absorbency of the blank solution from the absorbency of the standard solution; take the mass of plumbum as the abscissa and the corresponding absorbency as the ordinate to draw up the work curve.

A.9.4.3 Determination

Weigh about 5 g sample, accurate to 0.0002 g, put it into an Erlenmeyer flask, add in 50 mL Class II water, add in 1 mL nitric acid, place it onto an electric stove to be heated to slight boiling, when slightly cooled, it is filtered through moderate-speed qualitative filter paper and use Class II water to rinse it for three to five times, merge the rinsing solution and the filtrate into a 50 mL volumetric flask, dilute it with Class II water to the scale, shaking evenly.

A.9.5 **Result calculation**

The plumbum content is calculated by the mass fraction of w4 and the numerical value is indicated by mg/kg, which is calculated according to formula (A.4):

$$W4 = \frac{m1}{m \times 10\ 3 \times (100-w1)} \times 100\% \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (A.4)$$

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Where:

$m1$ = the numerical value of the mass of the plumbum in the testing solution checked form the work curve, in the unit of milligram (mg);

m = the numerical value of the mass of the sample, in the unit of gram (g);

$w1$= the mass fraction of the loss on drying determined according to A.4, the numerical value is indicated by %.

The arithmetic mean of the result of two measurements in the same condition is taken as the determined result, where the absolute difference between the results of two measurements in the same condition shall not be more than 2 mg\kg.

END TRANSLATION