

**Research and fabrication of toxic filter materials
from Tra Bac coconut shell activated carbon applied
in the manufacture of personal respiratory protection devices**

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ABSTRACT

This paper presents the results of research on making materials to adsorb toxic acidic gases, organic substances from Tra Bac coconut shell activated carbon, and combined with chemical treatment on the laboratory scale. Methods were used for this study consisting of SEM, EDS; BET, and DPC5 (kinetic adsorbent). The results showed that the fabricated filter material has a surface area of 664 m²/g, calculated according to BET, has a protection time with benzene vapor (L= 5 cm, Co(vapor) = 18 mg/L) of 56 minutes; protection time with HCl acid vapor (L= 5 cm, Co (vapor) = 1.1 mg/L) is 280 minutes. The results of the study have exhibited a good ability to filter benzene vapor and HCl vapor. This material has the potential for use in the manufacture of personal respiratory protective equipment.

Keywords: Activated carbon; Adsorption material; Respiratory protective equipment.

1. INTRODUCTION

A fire escape mask is a piece of personal equipment used to protect the human respiratory system from overcoming fire, smoke, and dust when a fire occurs. The mask has the effect of filtering harmful agents generated by the fire, such as CO, HCN (asphyxiation gas), HCl, HBr, HF, COF₂, H₃PO₄ SO₂, NO_x (eye irritation), and other organic irritants such as acid vapor, acrolein, formaldehyde, crotonaldehyde, phenol, styrene, and benzene, dioxin, carcinogens [1].

Dust particles (especially ultrafine particles) are particles of carbon black, fragments of organic hydrocarbons that undergo cyclic cycling, forming aromatic ring compounds that combine into sheets like graphite molecules. Besides, volatile toxic substances, including acids, organic irritants, and carcinogens, condense on the particles. These particles that are inhaled into the lungs will cause poisoning [2, 3].

In the filter box of the mask to overcome the fire, there are layers of materials like the paper layer has the effect of filtering dust and aerosols, and the additive-impregnated activated carbon layer has the effect of filtering organic vapors, acid vapor, and CO [4, 5].

The aim of this article is focused on the research and fabrication of materials that adsorb organic substances, acid vapors, and filtering dust and aerosols generated by fires.

2. PROBLEM

2.1. Methods of manufacturing adsorbent material

The method of filter layer fabrication includes the following steps:

Step 1: Preparation of materials

- Tra Bac coconut shell activated carbon TBA1, K₂CO₃ Pa mecrk, and additives;

- Oven;
- Rotary oven and some other tools.

The obtained activated carbon was determined to the specifications, then dried at 120 °C for 8 hours.

Step 2: Preparation of solution impregnated with K_2CO_3 and additives

K_2CO_3 solution and additives are prepared with concentrations of 5%, 10%, 15%, 20%, 25%.

Step 3: K_2CO_3 solution and additives were impregnated on activated carbon, then incubated for 24 hours and dried at 120 °C for 2 hours.

Step 4: Pyrolysis

Additive impregnated activated carbon is pyrolysis in a rotary kiln.

2.2. Determination of filterability of materials

Determine the protection time of the filter layer with benzene vapor and HCl vapor of the filter layer on the DPS5 dynamic adsorption device.

The methods of evaluating technical parameters include SEM-EDS, X-Ray, and N_2 adsorption isotherm measurement were used to determine BET surface area and porosity parameters.

3. RESULTS AND DISCUSSION

3.1. Research to determine the technical parameters of activated carbon

Measurement results of N_2 adsorption-desorption isotherm of Tra Bac coconut shell activated carbon is presented as follows. From measurement results of the N_2 adsorption-desorption isotherm and BET curve, the specific surface area is $1150 \text{ m}^2/\text{g}$, the small capillary volume is $0.46 \text{ cm}^3/\text{g}$, and the average capillary volume is $0.087 \text{ cm}^3/\text{g}$. Measurement and evaluation of technical parameters and a summary of the results of the evaluation of technical parameters of activated carbon samples are presented in table 1.

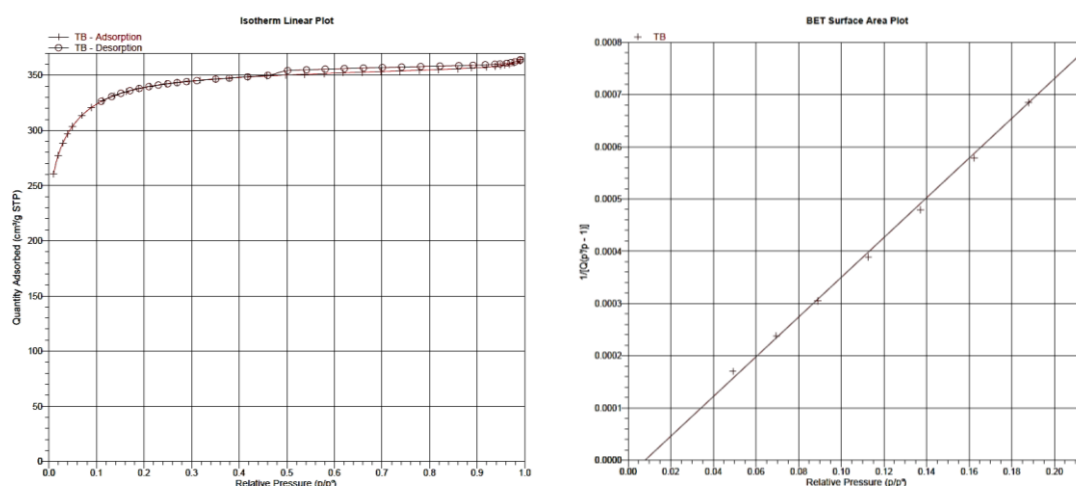


Figure 1. N_2 adsorption-desorption isotherms and BET curve of Tra Bac coconut shell activated carbon sample.

Table 1. Technical parameter measurement results of Tra Bac coconut shell activated carbon samples.

No.	Technical parameters	Measurement results
1	Morphology	Black, fine granular
2	Particle size, (mm)	1.0-1.5
3	Grain strength (%)	99.5
4	Specific gravity (g/cm ³)	0.47
5	Water absorption (%)	100
6	Small capillary volume (cm ³ /g)	0.46
7	Average capillary volume (cm ³ /g)	0.087
8	Surface area (m ² /g)	1150
9	Maximum adsorption capacity of benzene vapor P/P _s = 0.99 (mM/g)	6.15

The obtained results showed that Tra Bac coconut shell activated carbon sample has high particle strength, high specific surface area, and high benzene vapor adsorption capacity. Therefore, using coconut shell-activated carbon as a carrier to make materials to adsorb organic substances and some acidic toxins.

3.2. Study on the effect of K₂CO₃ impregnating agent content on the protection time of activated carbon impregnated with additives

A study on the effect of K₂CO₃ impregnating agent content on the protection time of additively impregnated activated carbon showed that the concentration of K₂CO₃ impregnating agent investigated was from 5% to 25%.

The protection time with HCl vapor of the catalytically impregnated activated carbon layer was performed on the DPC5 dynamic adsorption measuring device, with the measurement conditions including the thickness of the coal layer in the kinetic tube L = 5 cm, humidity 95%, gas flow rate 1.5 liters/min, hydrochloric acid vapor concentration 1.1 mg/L. The results are shown in table 2 as follows.

Table 2. Effect of K₂CO₃ impregnating agent content on protection time with HCl vapor of additively impregnated activated carbon.

No.	Impregnant content (%)	Protection time (minute)
1	5	96
2	10	198
3	15	280
4	20	296
5	25	285

From the data table, the results of measuring the protection time with HCl vapor of the activated carbon samples impregnated with the catalyst in table 2, a graph describing the dependence between the time of protection with HCl vapor on the impregnation content is shown in figure 2 below.

The results in figure 2 show that: When the impregnating content increases from 5 to 20%, the protection time with HCl vapor increases. In samples with 20% impregnated

content, the protection time was highest. When the impregnating content is increased above 20%, the protection time decreases, which indicates that the high impregnating content has the ability to block the porous structure of the activated carbon, so the protection time with acid vapor is reduced. On the other hand, after making the catalyst, the activated carbon sample impregnated with 20% K_2CO_3 has more dust than the sample impregnated with 15%. Samples of activated carbon impregnated with K_2CO_3 content higher than 15% have a white surface phenomenon. From the results of this study, a suitable 15% K_2CO_3 impregnating content was selected for catalyst fabrication.

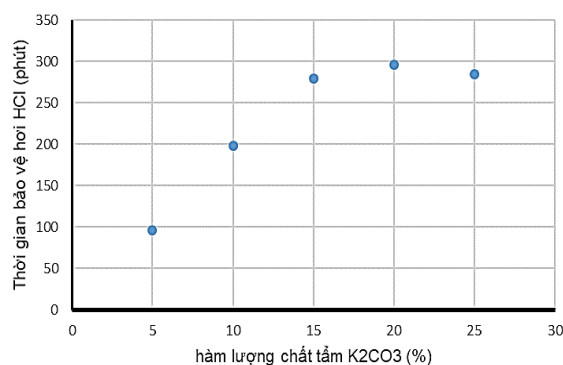


Figure 2. Graph of the dependence between HCl vapor protection time on the impregnation content of K_2CO_3 on activated carbon.

3.3. Evaluation of technical parameters of activated carbon samples impregnated with additives

The results of the analysis and evaluation of the properties of the activated carbon sample impregnated with the catalyst are shown below.

Take pictures on scanning electron microscope SEM to observe the surface of the catalytically impregnated activated carbon sample shown in figure 3 below.

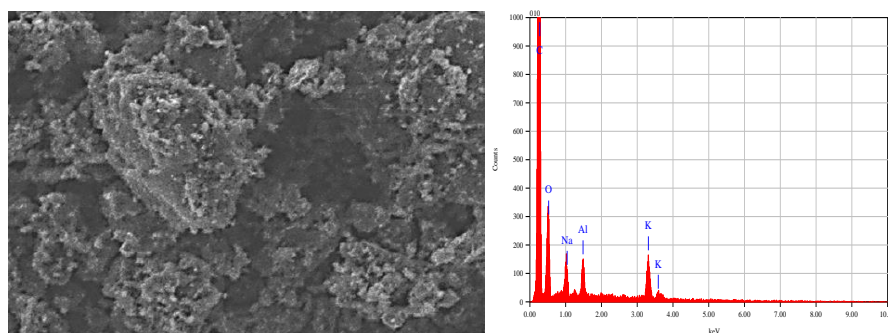


Figure 3. SEM image and EDS spectrum of activated carbon sample impregnated with catalyst.

The results of semi-quantitative analysis of the composition of the activated carbon sample impregnated with catalysis by EDS spectrum were given in figure 3, in the sample containing elements C, O, and K. Combined with the method of volumetric analysis, the exact quantification of K_2CO_3 composition is: 14.84%. This result is different from the calculated amount put into impregnation, which is $(15-14.84) * 100/15 = 1.06\%$.

Measurement results of N₂ adsorption-desorption isotherm of coconut shell activated carbon material impregnated with K₂CO₃ catalyst additive are shown in the following figure 4.

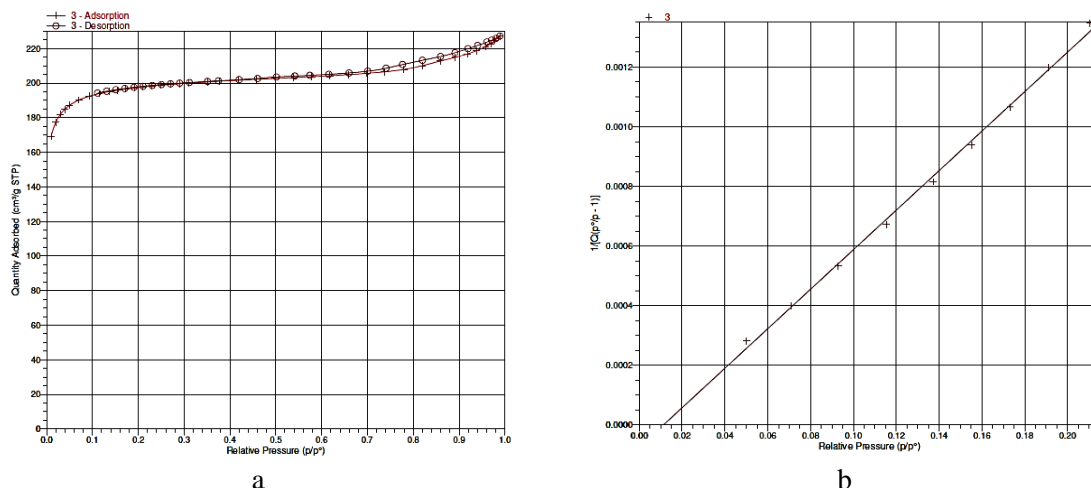


Figure 4. a - N₂ adsorption-desorption isotherm; b - BET surface area plot.

The above results determine the BET surface area of the catalytically impregnated activated carbon and the pore distribution in the activated carbon material. The results of the evaluation of the technical parameters of the activated carbon material impregnated with K₂CO₃ catalyst are defined in table 3.

Table 3. Technical specifications of activated carbon samples impregnated with catalysts and the original activated carbon sample.

No.	Technical parameters	Activated carbon Coconut shell	Coconut shell activated carbon with catalyst
1	Morphology	Black, granular	Black, granular
2	Particle size, (mm)	1.0-1.5	1.0 – 1.5
3	Grain strength (%)	99.5	99.5
4	Specific gravity (g/cm ³)	0.47	0.65
5	Small capillary volume (cm ³ /g)	0.460	0.360
6	Average capillary volume (cm ³ /g)	0.087	0.075
7	Surface area (m ² /g)	1150	664
8	Water absorption (%)	31	45
9	Maximum adsorption capacity of benzene vapor P/Ps = 0.99 (mM/g)	6.15	4.9

The results of determining the specifications of the sample of activated carbon impregnated with the catalyst compared with the original sample of coconut shell activated carbon have shown that: The sample impregnated with activated carbon has a good adsorption effect on HCl acid vapor and also good adsorption capacity of organic substances, high benzene vapor adsorption capacity. The results of this research and fabrication of this catalytically impregnated activated carbon sample can well meet the needs of users in the manufacture of filter cartridges and environmental treatment.

3.4. Test results for the acid vapor filtration ability of the material on the DPS - 5 dynamic adsorbent

Study on determination of protection time with HCl vapor of catalytically impregnated activated carbon layers used DPC5 dynamic adsorption device with measuring conditions: the thickness of catalytically impregnated activated carbon layers in kinetic tube different $L = 0.5$; first; 2; 3; 4; 5 cm; 95% humidity, gas flow rate 1.5 liters/min, HCl vapor flow concentration is 1.1 mg/l. The results are shown in table 4 as follows.

Table 4. The dependence of protection time with HCl vapor on the thickness of the activated carbon layer impregnated with catalysts.

No.	Coal layer thickness, cm	Protection time, minutes
1	0.5	18
2	1	32
3	2	91
4	3	151
5	4	218
6	5	280

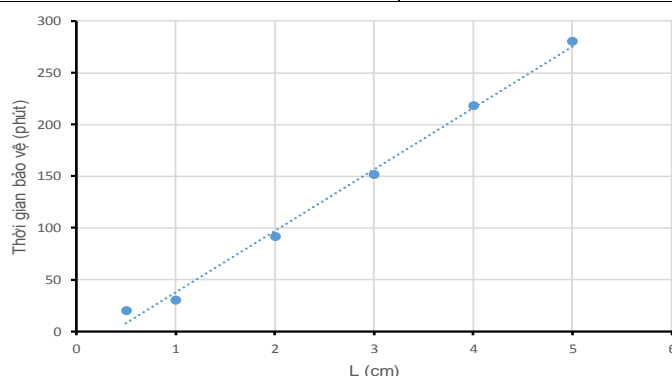


Figure 5. The dependence of the protection time of HCl acid vapor on the thickness of the layer of activated carbon impregnated with catalysts.

From the results in table 4, a graph describing the dependence between protection time on the thickness of the activated carbon layer impregnated with catalyst, the graph is shown in figure 5.

The test results on the experimental graph in figure 5 exhibited the kinematic parameters such as dead layer thickness $L = 0.3$ cm, non-working layer thickness $L = 0.4$ cm, and working layer thickness $L = 0.6$ cm. These kinetic parameters are used for designing and manufacturing filter cartridges.

Research to determine the protection time with benzene vapor of catalytically impregnated activated carbon layers used a dynamic adsorption device DPC5 with measuring conditions: the thickness of layers of catalytically impregnated activated carbon in the duct different learning $L = 0.5$; first; 2; 3; 4; 5 cm; humidity 95%, airflow rate 1.5 liters/min, airflow concentration 18 mg/l. The results are shown in table 5 as follows.

From the results in table 5, a graph describing the dependence between the protection time of benzene vapor on the thickness of the activated carbon layer impregnated with the catalyst, the graph is shown in figure 6.

The obtained results on the experimental graph in figure 6 the kinetic parameters such as dead layer thickness $L = 0.4$ cm, non-working layer thickness $L = 0.5$ cm, and working layer thickness $L = 0.6$ cm. These kinetic parameters were used for filter box design and fabrication.

Table 5. Dependence of time of protection against benzene vapor on the thickness of the activated carbon layer impregnated with catalysts.

Nr	Coal layer thickness, cm	Protection time, minutes
1	0,5	1
2	1	4
3	2	16
4	3	29
5	4	40
6	5	56

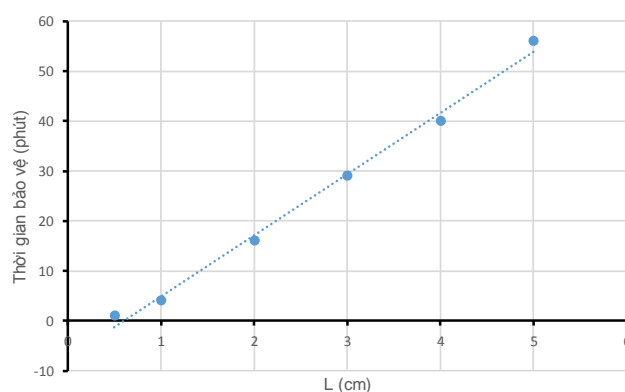


Figure 6. Protection time with benzene vapor depends on the thickness of the layer of activated carbon impregnated with catalysts.

4. CONCLUSIONS

Evaluation of technical specifications of activated carbon samples available in the laboratory scale for the selected Tra Bac coconut shell activated carbon sample was based on the technical criteria such as high benzene vapor adsorption capacity, surface area, large pore volume, high particle strength. These criteria selected activated carbon as a carrier in the research and manufacture of catalysts.

By investigating influencing factors in the process of building technology to make activated carbon impregnated with K_2CO_3 catalyst; the appropriate impregnated K_2CO_3 content was obtained as 15%.

Fabrication and evaluation of technical specifications of activated carbon material impregnated with K_2CO_3 catalyst showed that the material has good filtering ability of acidic toxic substances, and at the same time has a good adsorption capacity of organic substances, due to having a large surface area, high pore volume, high grain strength.

Tested to determine the protection time with HCl vapor and benzene vapor of catalytically impregnated activated carbon used the dynamic adsorption device DPC5, with different thicknesses, $L = 0.5, 1, 2, 3, 4, 5$ cm. From there, an experimental line graph describing the dependence between protection time and coal layer thickness $t = f(L)$

(Sylop experimental line). Where is the development direction of the research problem, and can it be further developed or not.

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REFERENCES

- [1]. Nguyen Dinh Hoa, Nguyen Hung Phong, Bui Van Tai (2003), "Study on impregnating Cu, Cr, Ag catalyst additives on activated carbon fabric used as a filter material for toxic gases". Research conference on prevention of nuclear, biological and chemical weapons, Center for Science, Technology and Technology, pp. 266-271.
- [2]. Nguyen Hung Phong, Le Xuan Tuan, Bui Van Tai (2003), "Research on preparation of carbon fiber adsorbents", 4th National Collection of Chemical Scientific Reports, Hanoi, p. 81-86.
- [3]. Nguyen Hung Phong (2005), "Activated carbon impregnated with additives, catalysts used in the field of toxic filtration and environmental treatment in Vietnam". Collection of scientific reports at the 5th National Conference on Catalytic Adsorption, pp. 114-123.
- [4]. Nguyen Hung Phong, Nguyen Dinh Hoa (2005), "Preparation of acid-absorbing materials on the basis of activated carbon cloth". Collection of Scientific Reports of the 3rd National Conference on Catalysis - Adsorption, p. 131-136.
- [5]. Abdi Atilgan, Huseyin Peker and Hatice Ulusoy (2012), "Effects of different impregnation chemicals on combustion characteristics and decay resistance of wood", International Journal of Physical Sciences, Vol. 7, No. 47, pp. 6149-6157A.

TÓM TẮT

Nghiên cứu và chế tạo vật liệu lọc độc từ vỏ dứa Trà Bắc ứng dụng trong sản xuất thiết bị bảo vệ hô hấp cá nhân

Bài báo này trình bày kết quả nghiên cứu chế tạo vật liệu hấp phụ các loại hơi khí độc mang tính axit, các loại chất hữu cơ từ than hoạt tính gáo dừa Trà Bắc và hóa chất trong phòng thí nghiệm. Phương pháp sử dụng: SEM, EDS; BET; xác định thời gian bảo vệ với hơi benzen và HCl trên thiết bị hấp phụ động lực học DPC5,... Kết quả chỉ ra rằng, vật liệu lọc chế tạo được có diện tích bề mặt tính theo BET đạt 664 m²/g, có thời gian bảo vệ với hơi benzen (L = 5 cm, Co = 18 mg/l) là 56 phút; thời gian bảo vệ với hơi axit HCl (L = 5 cm, Co = 1,1 mg/l) là 280 phút. Vật liệu có khả năng sử dụng trong chế tạo các phương tiện bảo vệ hô hấp cá nhân.

Từ khoá: Cacbon hoạt tính; Than hoạt tính.