Available online at www.sciencedirect.com

SciVerse ScienceDirect

Energy Procedia 18 (2012) 1145 – 1151

Energy Procedia

Preparation and characterisation of an natural adsorbent used for elimination of pollutants in wastewater

ARRIS Sihem*, BENCHEIKH LEHOCINE M and, MINIAI H.A

Laboratory of the engineering and the processes of environment (LIPE)
Department of industrial chemistry, Faculty of Science of the engineer, University of Constantine, 25000, Algeria.
E-mail: arris_s@yahoo.fr

Abstract

In recent years the uses of waste as a raw material for producting porous carbon have been widely employed in the purification of water and wastewater. The application of these carbons has bees considered also a major unit operation in the chemical and petrochemical industries. The purpose of this study is to characterize a natural adsorbent obtained from local cereals by products. This solid has been tested with organic pollutants (phenol, dyes) and inorganic pollutants (copper, zinc and cadmium) where he was given a good performance. The characterization is important to understand and identify the different phenomena of retention (adsorbent-adsorbate) as well as to the interpretation of the kinetic results. The characterization is focused on quantitative analysis (elemental analysis, BET), qualitative (RX, IR) and optical (SEM) in order to get an idea on the morphology and pore structure and active sites.

Keywords: Characterization of adsorbents-methods qualitative -quantitative methods-optical methods-elemental analysis- BET- SEM-IR-RX

1. Introduction

Adsorption is a well established and power full technique for the removal of organic and inorganic pollutants from waste waters. Activated carbon is widely used as adsorbent in gas and liquid phase separation. In addition to serving as an adsorbent, high porosity carbons have recently been applied in the manufacture of high-performance layer capacitors. Because of the introduction of rigorous environmental regulations and the development of new applications, the demand for porous carbons is expected to increase progressively [1].

Precursor materials for the commercial activated carbons are coal, lignite, nutshells, wood and peat, which are transformed in carbonaceous materials by proper carbonization [2]. Typical activated carbons consist of particles with porous structure having a network of interconnected macropores, mesopores and

1876-6102 © 2012 Published by Elsevier Ltd. Selection and/or peer review under responsibility of The TerraGreen Society.
Open access under CC BY-NC-ND license. doi:10.1016/j.egypro.2012.05.129

micropores resulting to high surface area and good capacity, ionic nature and functional groups determine the nature of bonding mechanisms and the extent of adsorption [3].

The aim of this paper is to characterize a cereal by-product which has a very abundant production in Algeria. This solid material is used as a natural adsorbent for removal organic and inorganic pollutants.

2. Characterization of adsorbent:

2.1. Preparation of adsorbent:

The agricultural by-products were:

- washed;
- Calcined at a temperature of 600C in exclusion of air in an electrical furnace (HEARAEUS D-6450 HANAU/Germany);
- Crushed in a crusher (FRITSCH industry.8 6580 Idar Oberstein);
- Separated in 3 different size classes :G1, G2, G3 respectively : lower than 0.1mm, 0.3mm, 1mm by using a sifter (Analysensieb-retsch-5657 HAAN W);
- Finally, the solid material obtained is stored in a desiccator for use.

Before calcinations the adsorbent is called DCB

After calcinations the adsorbent is called DCC

2.2. Elemental analysis:

This analysis was carried out at the National center of the Scientific research in France (CNRS) at the central service of analysis. The results obtained are shown on the following table:

Table 1: Results of elemental analysis

(%)	C	H	N	O	S	Cl	Mg	Na	K	Ca	Cu
DCB	41.24	1.14	2.85	24.25	0.30	≤0.20	2.40	0.25	5.21	0.97	143
(%)	C	H	N	O	S	Cl	Mg	Na	K	Ca	Cu
DCC	65.76	1.64	4.41	22.02	0.21	≤0.20	0.59	0.12	0.67	0.67	865

The composition of our support is given by mass percentages. According to the values represented on table 1, it is seen that the cereal scrap mainly supports carbon and oxygen. This leaves as to conclude the existence offunctions oxides. Because of the high proportion of carbon we can say that the cereal by product can be an activated carbon after some operations.

2.3 The measurement of specific surface:

The analyzes concerning the measurement of specific surface (BET) were carried out in the Research center and Developpement CRD, direction Sonatrach (BOUMERDES – Algeria). All the results obtained are represented below

Table 2: Results of BET

Support	DCB	DCC
S (m^2/g)	0	54.7

The values obtained indicate the existence of macrospores on the surface of the cereal by products.

2.4 Analysis of cereal by-products on the SEM

The analysis of our solid material prepared at the laboratory under the electron microscope was carried out at the Organic and Macromolecular Chemistry laboratory at the University of Technological Sciences of LILLE. Scanning electron microscopy is a technique of electron microscopy based on the principle of electron-matter interactions, capable of producing high-resolution images of the surface of a sample. The images collected are represented below:

a-For DCB

The four following images give the morphology of the cereal scrap at the rough state (without any preliminary treatment).

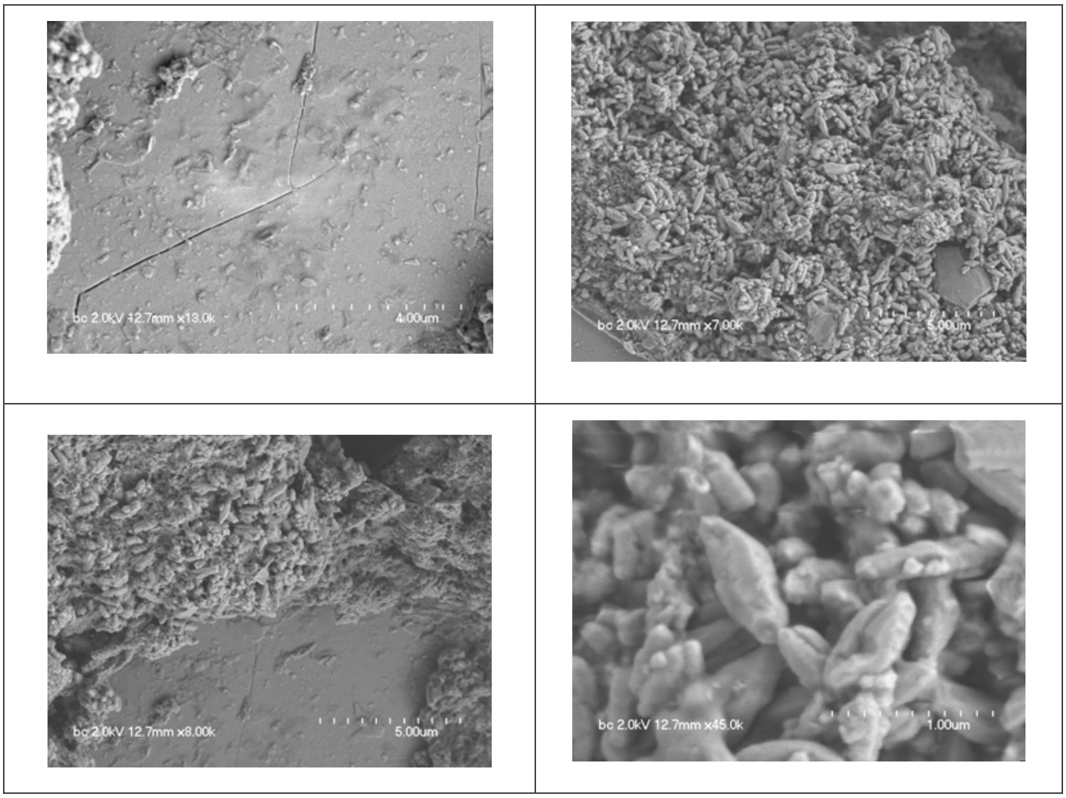

Figure 1: Board of photographs of the DCB to the SEM

b- For DCC:

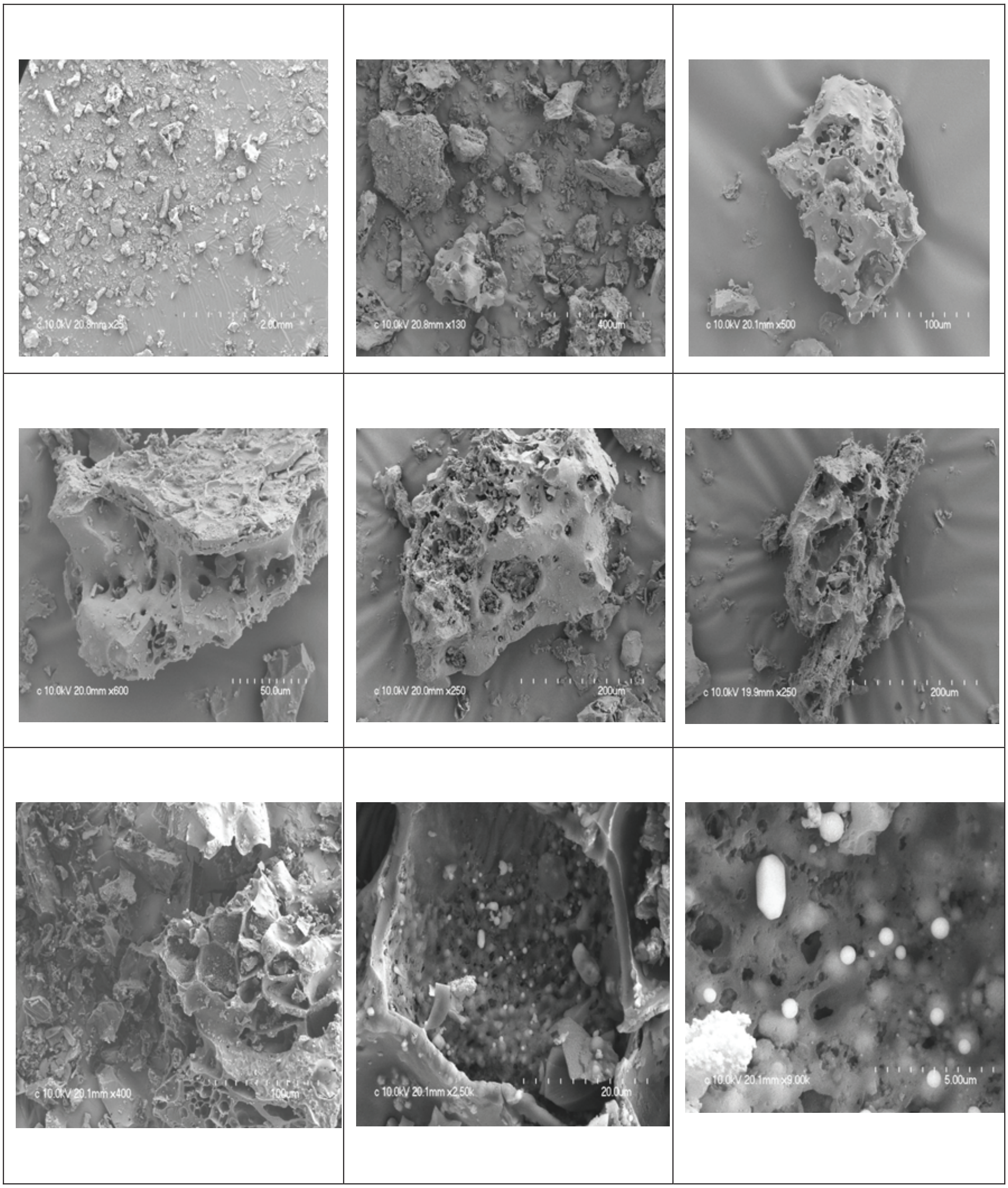

Figure 2: Board of photography of the DCC to the SEM

This board of photography shows that the grains of the DCB are more or less homogeneous. They have the shape of channels closed at the two ends. The agglomerates of the particles meet between them by creating a fitting. This last is the person in charge of the determination of the porosity of the fragments. The vacuums or interstices left between the particles form the pores.

The observation of the DCC under the electron microscope shows a polydispersity in the face and heterogeneity of the porous structure. If one observes one of the fragments formed after the calcinations, we can notice that it presents a porous form. Thus in addition to the pores which exist between the fragments, they exist other pores which are located on these fragment.

The examination pushed on the pores existing on the fragments shows the continuity of the cavities within the pores. These cavities are more or less heterogeneous from one pore to another and fragment to another.

2.5 Analysis by IR:

The analysis of our solid adsorbent prepared at the laboratory by wills infra red was carried out at the Organic and Macromolecular Chemistry laboratory at the university of technological sciences of LILLE. The spectrum obtained is represented below:

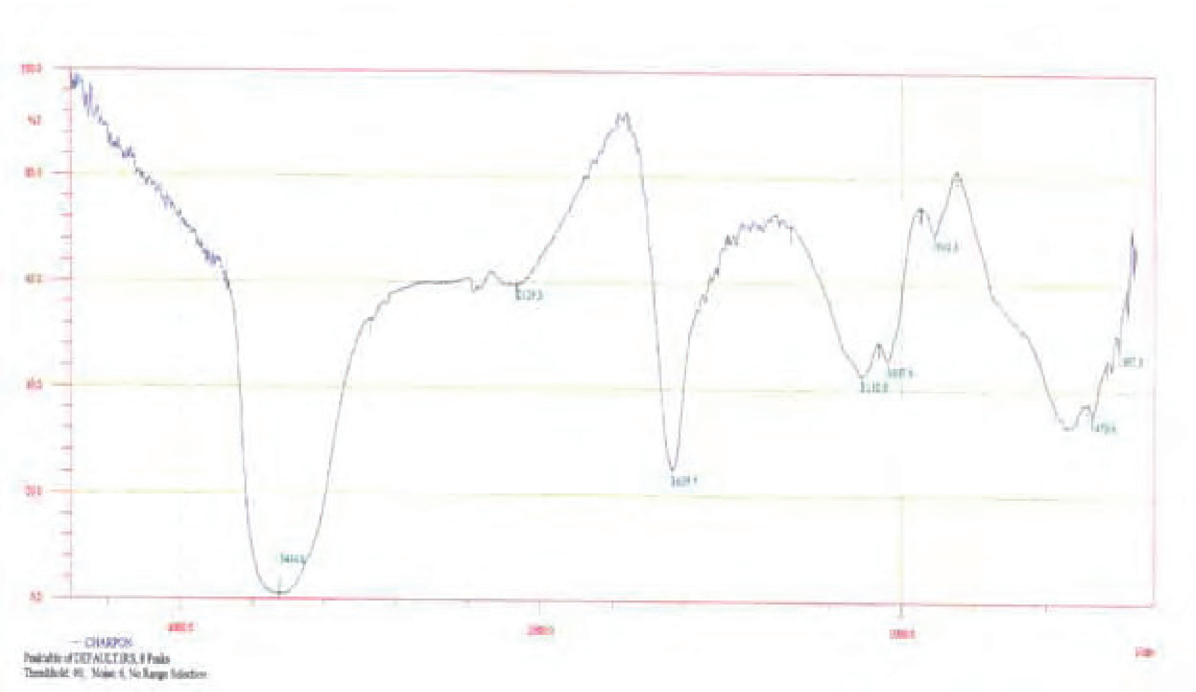

Figure 3: Spectrum of the IR of the DCC

The spectrum shows a band of adsorption to 1631.7 cm-1 indicating the presence of grouping C=O of which the configuration quinone [4]; and a band to 3467.8 cm-1 corresponds to the vibration of function OH [5]. This conclusion coincides with our results obtained for the ultimate analysis, which confirm the existence of the surface functions including/understanding of oxygen.

2.6 Analysis by RX:

The analysis of our solid support prepared at the laboratory by X-ray was carried out at the Organic and Macromolecular Chemistry laboratory at the university of technological sciences of LILLE. The spectrum obtained is represented below:

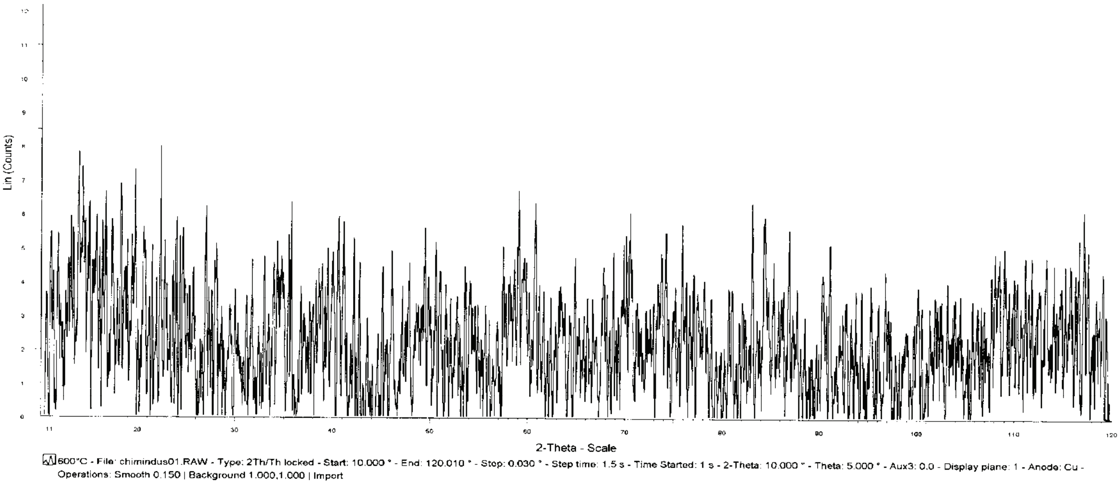

Figure 4: Spectrum of the X-Ray of the DCC

The superposition of the spectrum obtained with standardized cards ASTM indicated the presence of the elements according to: C, O, S, K, Na, Ca, Mg. A proportioning carried out on our support carried out in a study proposed by N.GHERBI [6] (on the same support) watch the presence of the cations (Na^+, K^+, Mg^{+2}, Ca^{+2}) on the surface of coal what informs us that certain functions of surface have the alkaline salt shape RO-Na+ [7].

3. Conclusion and prospects

The results obtained show a heterogeneous structure presenting of the macro pores on certain fragments, what confirms the moderate value of the specific area. Elemental analysis showed the presence of the elements carbon, hydrogen, nitrogen and oxygen in the structure of adsorbent. This prediction is confirmed by IR analysis which shown the presence of two principals functions: when we have a pick at 1631.7 cm-1, indicating the presence of grouping C=O ; and a band at 3467.8 cm-1 corresponds to the vibration of function OH.

.The SEM analysis of cereal by-product gives two boards of photography: the first one for the natural solid without any treatment (DCB) and the second for the solid after calcinations (DCC).

The grains of DCB are more or less homogeneous. They have the shape of channels closed at the two ends. For DCC, we can see clearly that the porosity is developed across the sample surface with a polydispersity in the face and heterogeneity of the porous structure. If one observes one of the fragments formed after the calcinations, we can notice that it presents a porous form. The examination pushed on the pores existing on the fragments shows the continuity of the cavities within the pores. Which indicate the presence of micro pores.

We can see also that the surface area is better when the sorbent was carbonized (0 and 54,7m^2/g). This is appears clearly in the photographys befor and after calnination.

The spectrum of X-Ray confirms the existence of function surface contaning oxygen. This constatation is validates with the results of elemental analysis.

This study is only one attempt which had like objective; to test the cereal by-products as an adsorbent using for the depollution of water. The cereal by product used in this study has not any treatment of preliminary activation. In the future we propose an experimental study of physical and chemical activation of the DCC with an aim of improving his effectiveness.

Bibliographical references:

[1] Kirubakaran, C.J, Krishnaiah, K, Seshadri, S.K. Ind. Eng. Chem. Res. 1991, 30, 2411-2416.

[2] Sircar, S., Golden, T. C, Rao, M.B/ Carbon 1996, 34, 1-12

[3] N. Calace, E. Nardi, B.M. Petronio and M. Pietroletti, Environ. Poll., 118 (2002) 315-319.

[4] J.F. Patterson, Industrial Wastewater Treatment Technology, 2nd ed.; Butterworths, London, 1985

[5]N. Gherbi, A-H. Meniai, B. Bencheikh lehocine. Desalination. 166 (2004) 363-369.

[6] G.SCACCHI, M.BOUCHY, J.F.FOUCAUT, O.ZAHAAA 1996 ‘Cinétique ET Catalyse’ Génie des procédés de l’école de Nancy.Tec et Doc,

[7] W. J. WEBER, P. M. Mc GINLEY, L. E. KTZ 1991 ‘Sorption in subsurface systems concept, models and effects on contaminant fate and transport. Water Research, Vol. 25, p.499-528,

[8] E.L. SLEJKO, M.DEKKER 1985 ‘Adsorption Technology: a step by step approach to process evaluation and application, Ed M.DKKER, New York

[9]A.L. Ahmad*, S. Bhatia, N. Ibrahim and S. Sumathi July - September, 2005‘ADSORPTION OF RESIDUAL OIL FROM PALM OIL MILL EFFLUENT USING RUBBER POWDER’ Brazilian Journal of Chemical Engineering Vol. 22, No. 03, pp. 371 - 379,

[10]S.ARRIS, M. Bencheikh-Lehocine, A.H.MENIAI M.Morcelet, M. Bacquet, B. Martel, and A.Mansri, 2003 “Study and Identification of Retention process of heavy Metals by Adsorption on Agricultural By-products”. Chemical Engineering and Technology’, ISSN 0930-7516 CETEER 26 (9) 2003 919-1018 Vol.26-No.9 September.

[11] Marı´a Luisa Soto, Andre´s Moure *, Herminia Domı´nguez, Juan Carlos Parajo 2008 ‘Charcoal adsorption of phenolic compounds present in distilled grape pomace’ Journal of Food Engineering 84 (2008) 156–163.

[12] Ahmet O¨ rnek a, Mahmut O¨ zacar b,∗, I˙. Ayhan S¸ engil 2007 ‘Adsorption of lead onto formaldehyde or sulphuric acid treated acorn waste: Equilibrium and kinetic studies’ Biochemical Engineering Journal 37, 192–200, 2007

[13]A.L. Ahmad*, S. Bhatia, N. Ibrahim and S. Sumathi , July - September, 2005 ‘ADSORPTION OF RESIDUAL OIL FROM PALM OIL MILL EFFLUENT USING RUBBER POWDER’ Brazilian Journal of Chemical Engineering Vol. 22, No. 03, pp. 371 – 379.

[14]Vandana Singh a,∗, Stuti Tiwari a, Ajit Kumar Sharma a, Rashmi Sanghi 2007 ‘Removal of lead from aqueous solutions using Cassia grandis seed,gum-graft-poly(methylmethacrylate)’ Journal of Colloid and Interface Science 316 (2007) 224–232.