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Improvement Of The Rice Husk Based Activated Carbon By Utilizing Of Alkali Activation Technique



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ABSTRACT

Rice husk, as an agricultural waste, was utilized in production of high quality activated carbon by alkali chemical activation method. The process takes place through three steps, carbonization, activation and washing. Different operating parameters in each stage have been studied. The high quality activated carbons produced at optimum operating conditions have surface areas of 2470, 2980 m²/g and methylene blue values of 750, 940 mg/g for KOH and NaOH respectively.

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KEYWORDS

Activated carbon;
 Rice husk;
 Alkali activation.

INTRODUCTION

Activated carbon is the common term used for a group of porous carbonaceous material. It's most important property is the very large internal surface area which generated from high degree of porous crystalline structure^[9]. Such a surface area ranged from 600-1200 m²/g for commercial grade and reach to more over 3000 m²/g in advanced grades by using of modern activation methods like alkali activation technique^[3,6,13,17].

Such a high surface area of activated carbon is reflected in the large adsorptive power for many types of contaminations (color, smell, taste, heavy metals... etc). As a result, activated carbon is considered the most popular and cheapest material used in adsorption treatment applications.

Every year, approximately one hundred fifty thousand metric tons of powdered activated carbon is manufactured, together with one hundred fifty thousands metric tons of granular activated carbon and thirty thousands metric tons of pellets^[12]. Acti-

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ated carbons are prepared by simultaneous or separated carbonization and activation of different starting carbonaceous material (coals, polymers, wood, agricultural waste ... etc), using of agricultural waste as precursors for activated carbon production is greatly investigated nowadays because it is cheap and available in large quantity especially in the developing countries due to the large dependence of these countries on agricultural products. Numerous investigations have reported their studies by different traditional activation method which include physical activation with steam or carbon dioxide gas (CO_2) and chemical activation with zinc chloride (ZnCl_2) or phosphoric acid (H_3PO_4)^[4,8,10,11]. Where the activated carbon prepared by these methods exhibited low characteristics (low surface area, low yield and low mechanical properties). Recently, the production of activated carbon from rice husk by alkali activation technique has become a research topic of a great interest due to the enhanced characteristics of the produced activated carbon compared to other traditional activation methods^[2,15,16]. The present work investigates the production of a high quality activated carbon from rice husk by alkali chemical activation technique with potassium or sodium hydroxide.

EXPERIMENTAL

Rice husk used is obtained from MECCA Co. for rice milling, Tanta which is collected from different rice farms. Its analysis is shown in TABLE (1).

TABLE 1: Rice husk proximate and ultimate analysis

Proximate analysis (wt %)		Ultimate analysis (wt %)	
Volatiles	55	Carbon	44.9
Moisture	3-8	Hydrogen	5.6
Ash	15-17	Oxygen	49.3
Fixed carbon	22-25	Nitrogen	Nil

Experimental procedure

It is done in three steps; carbonization, activation and washing figure 1.

Rice husk is washed and fractionated to remove any entrained dust or clay then dried at 110 °C over-

night. 200 gm of the dried sample is fed to a tube furnace; and carbonized at known temperature (300-600°C) and time (0-3hr) in presence of inert gas flow (N_2) to avoid the combustion of the sample. After the pyrolysis step is finished, the tube furnace is left to cool then; rice husk char is withdrawn and cooled in desiccators to room temperature. The sample is then activated under constant activation conditions and tested to study the effect of different parameters controlling the carbonization process.

Then, 10 gm of dried sample is mixed with a certain amount of KOH or NaOH (impregnation ratio 1:1-6:1) in a ceramic crucible and calcined in a muffle furnace from 600-900 °C for known time (0-3 hr) in absence of air. The calcined sample is then cooled and washed to obtain the required porous activated carbon.

Analysis of activated carbon

a. Methylene blue value

The methylene blue value was measured by spectrophotometry. About 0.1 grams sample of activated carbon was placed in a stopper glass flask. 100 ml of methylene blue solution of 1000 mg/l was added to the sample and shaken for 24hr. Then methylene blue is filtered and the concentration of the final was measured. The methylene blue value is expressed as mg/g.

b. Surface area

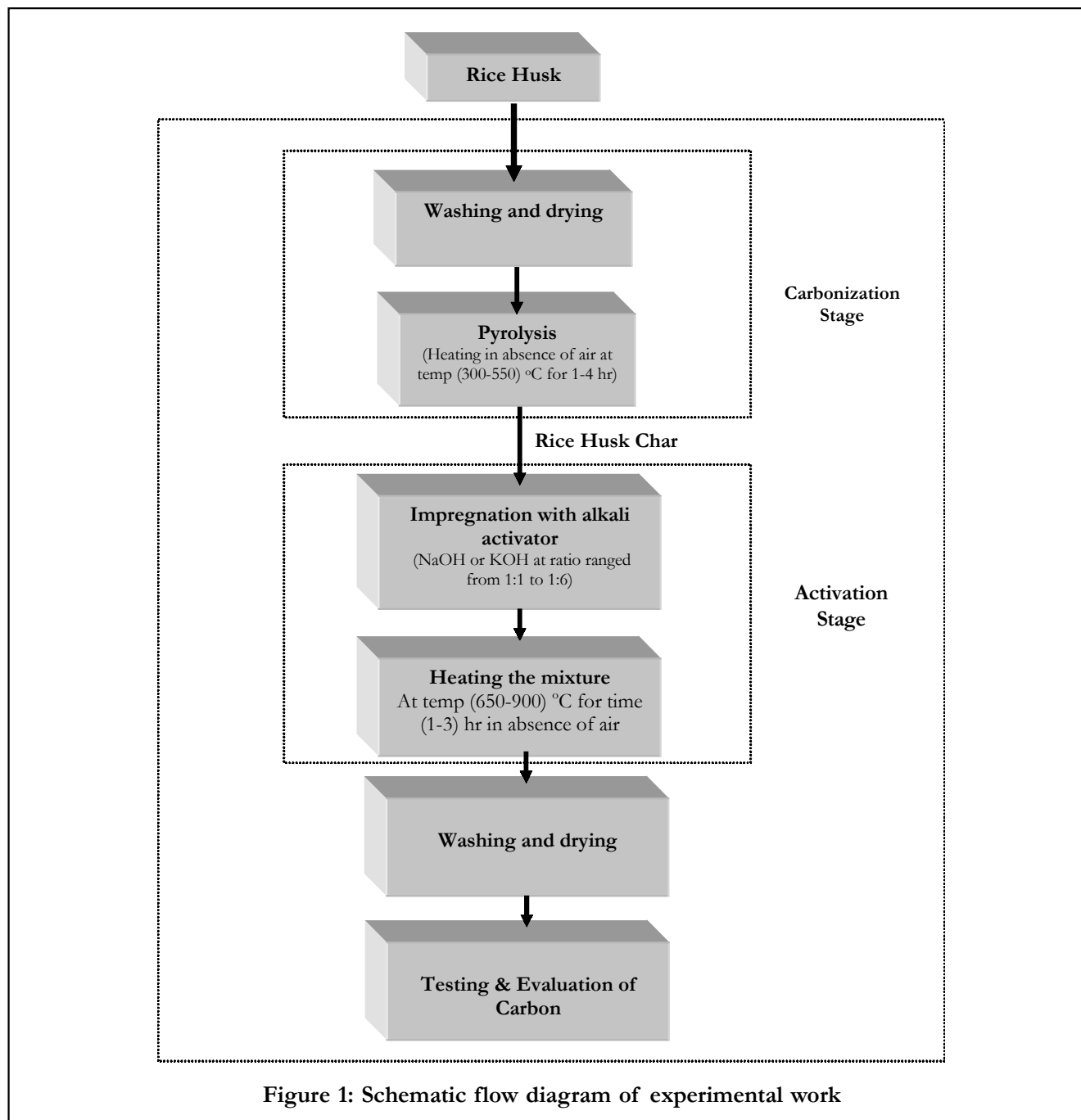
The surface area was determined by the B.E.T. method using the Quanta chrome NOVA Automated Gas Sorption System. The volume of adsorbed gas is calculated by measuring the pressure change resulting from adsorption of known volume of gas.

RESULTS AND DISCUSSION

Carbonization stage

1. Effect of carbonization temperature

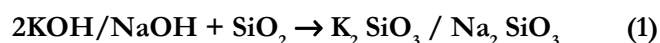
The adsorption capacity increases up to 680 mg/g with the increasing of carbonization temperature from 300°C to 450°C, figure 2; then it remains approximately constant by further increasing in carbonization temperature. This can be explained by silica distribution in rice husk, it is classified to internal



silica which distributed homogeneously within the lignocelluloses structure and external silica which distributed under the husk's outer surface^[4,5]. Thus, for rice husk char (non-activated carbon), increasing of carbonization temperature works to transfer the outer silica layer from amorphous state to crystalline state which form a glassy layer around the carbon particles which decrease the adsorption capacity by further increasing in temperature.

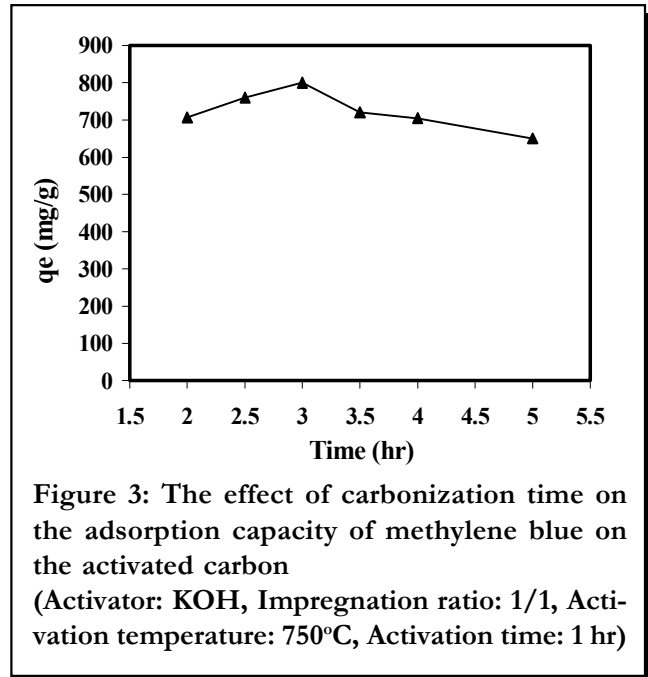
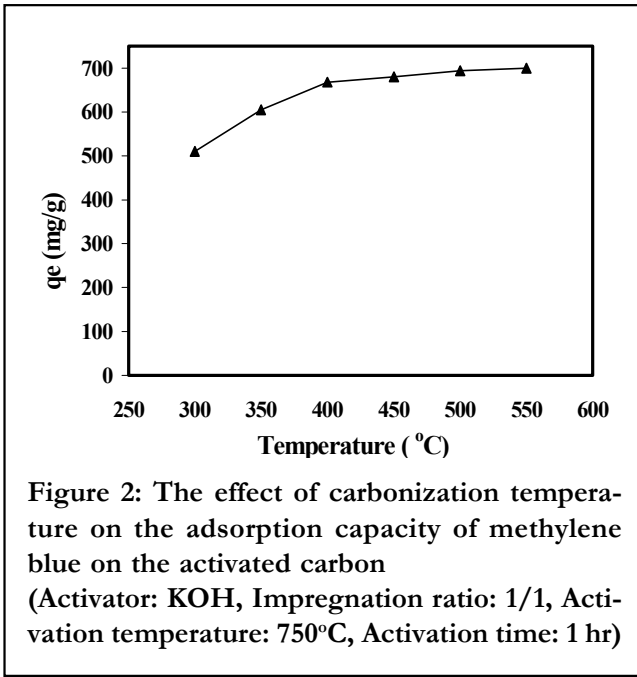
But, for activated carbon, alkali chemical acti-

vators (KOH or NaOH) react with internal and external silica content in rice husk (Equation 1) and remove the glassy layer formed around the carbon particles as^[15].



So, as the effect of silica has been excluded, the increasing in carbonization temperature work to enhance the graphitic crystalline structure that formed during the carbonization process, which increases the

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efficiency of final activated carbon.

2. Effect of carbonization time

The absorption capacity increases with time up to 3hr then decreases by further increasing in temperature (Figure 3). This may explained also as mentioned above by silica content in rice husk, which is distributed within lingo-celluloses structure, as it is transformed from amorphous to hard crystalline state which interferes with the graphitic structure of rice husk char. Thus, it destroys the uniform crystalline structure of carbon which reduces the efficiency of activated carbon due to the fact that the uniform graphitic structure of carbon is considered as an important factor in increasing of activation process efficiency^[9]. The yield of rice husk char produced after carbonization is found to be about 40% of the starting raw rice husk. TABLE 2 illustrates the elemental analysis of rice husk char.

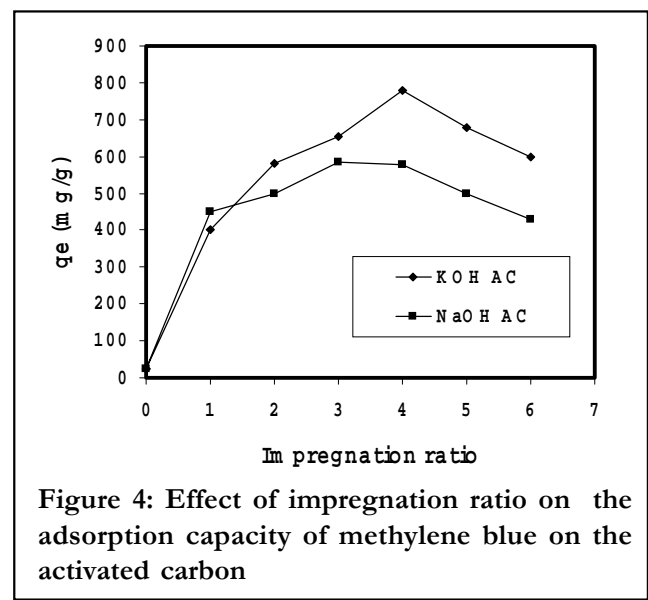
TABLE 2: Elemental analysis of rice husk char

Element	%
C	50
H	3.1
N	1.4
Ash (90% SiO ₂)	46

Activation process

1. Effect of impregnation ratio (Activator/char)

The effect of impregnation ratio has been studied at constant activation temperature and time respectively (800 °C and 1 hr), figure 4. It has found that the adsorption capacity increases with the increase of the ratio of both KOH/C up to 4/1 and NaOH/C up to 3/1. This indicate that increasing the activator ratio leads to excessive reaction on the activated carbon surface which increase the size of pores and hence decrease the adsorption capacity of



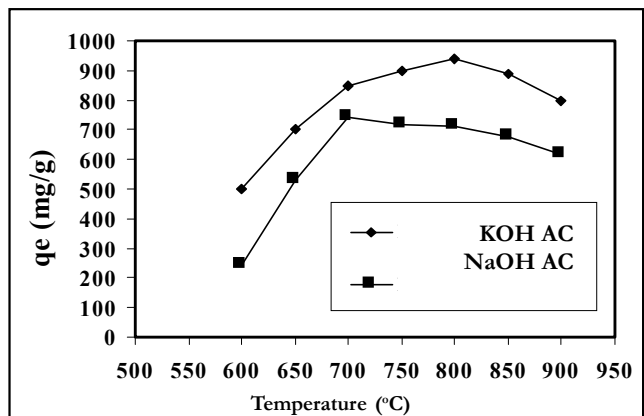


Figure 5: Effect of activation temperature on the adsorption capacity of methylene blue on the activated carbon

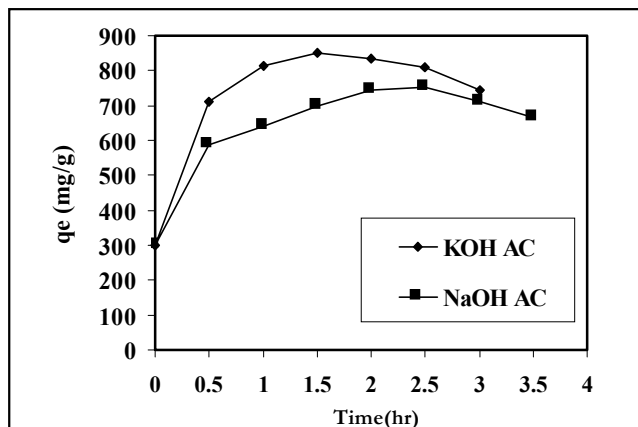


Figure 6: Effect of activation time on the adsorption capacity of methylene blue on the activated carbon

the carbon^[5].

2. Effect of activation temperature and time

The effect of activation time and temperature was studied at optimum impregnation ratios as shown in figures (5, 6). It appears that time and temperature has similar effect on the course of activation which may be explained on the fact that during the activation process the formation of pore is simultaneous with the destruction of pore^[5,9], so when the temperature and time are lower than 750°C and 1.5 hr the activation reaction increases with the increase of temperature and a large number of pores are formed, so the adsorption capacity increases. But when the temperature and time are higher than 800°C and 2 hr, the destruction of pores plays a leading role, so the adsorption capacity decreases gradually.

Washing process

1. Effect of different washing techniques

Different washing techniques at optimum impregnation ratios, activation time and temperature have been studied as shown in TABLE 3: It is clear

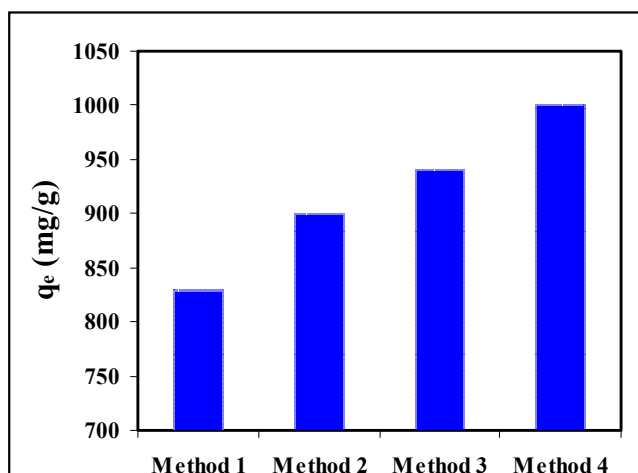


Figure 7: Effect of washing technique on the adsorption capacity of methylene blue on the activated carbon

that washing with water acidified with H₃PO₄ has the best washing efficiency (Figure 7), because H₃PO₄ has stronger acidity than HCl, so it has higher ability to dissolve any residues of carbonates salts which formed during activation course. Also, it dissolves any residues of silica in the activated carbon which

TABLE 3: Different washing techniques

Method 1	Washing with hot water till pH about 8
Method 2	Washing with hot distilled water till pH about 7.5
Method 3	1 st stage Washing with hot water
	2 nd stage Washing with hot acidified water (1% HCl)
	3 rd stage Rewashing with hot water to adjust pH to about 7
Method 4	HCl is replaced by H ₃ PO ₄ in method 3

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enhance the efficiency of the activation. But the selection between the third and the fourth methods depends on economic considerations.

For example, when applying alkali activation technique for the production of activated carbon from coals, washing with diluted HCl, is preferred because HCl solution has the ability to remove any sulphur contaminations, which may poison the activated carbon surface^[6].

Product evaluation

1. Methylene blue value

Figures (3-5) show that the methylene blue value reached the maximum (940mg/g) at 800°C, 1.5hr and KOH / C=4/1 in case of KOH, and (750 mg/g) at 700°C, 2 hr and NaOH / C=3/1 in case of NaOH.

2. Surface area

A surface area of activated carbon was determined by BET method using KOH and NaOH at the optimum conditions. It was found that specific surface area is 2962 m² and 2450 m² for KOH and NaOH respectively. It may be explained that reaction between NaOH and carbon surface begin at 570°C, where for KOH it is around 400°C. So, the longer activation time for KOH which start at 400 °C may increase the degree of activation^[3]. Also, the boiling point of potassium is 758°C which is lower than that of sodium 883°C, so, the potassium can enter into the interior of carbon structure and makes the activation freely while the outer activation of sodium hydroxide is stronger than potassium hydroxide^[6].

CONCLUSION

Carbonization temperature and time greatly influenced the carbon content and yield of char.

400°C temperature and 3 hr time were the optimum operating conditions for the carbonization steps. 40% yield was achieved under these conditions.

The optimum operating conditions for activation step were: 1.5hr, 750°C and 4/1 (w/w) impregnation ratio, in case of KOH and 1hr, 800°C, and 3/1 (w/w) impregnation ratio, in case of NaOH.

Activated carbon products obtained have meth-

ylene blue of 750mg/g and 940mg/g for NaOH and KOH respectively.

Surface areas of activated carbon products were 2980 m²/g and 2470m²/g for KOH and NaOH respectively.

REFERENCES

- [1] Byung-Dae Park, Seung Gon Wi, Kwang Ho Lee, Adya P. Singh, Tae-Ho Yoon, Yoon Soo Kim; *Biomass and Bioenergy*, **25**, 319-327 (2003).
- [2] Cecile F. Alfonso; *Philippine Technology Journal*, **21**, 5-26 (1998).
- [3] E. Raymundo-Piñeroa, D. Cazorla-Amorós, A. Linares-Solano, S. Delpeuxb, E. Frackowiakc, K. Szostakb, F. Béguinb; *Carbon*, **40**, 1614-1617 (2002).
- [4] Lotfi Monser, Nafaâ Adhou; *Separation and Purification Technology*, **26**, 137-146 (2002).
- [5] M.A. Lillo-Ródenas, D. Cazorla-Amorós, A. Linares-Solano; *Carbon*, **41**, 267-275 (2003).
- [6] M.A. Lillo-Ródenas, D. Lozano-Castelló, D. Cazorla-Amorós, A. Linares-Solano; *Carbon*, **39**, 741-739 (2001).
- [7] M.A. Lillo-Ródenas, D. Lozano-Castelló, D. Cazorla-Amorós, A. Linares-Solano; *Carbon*, **39**, 751-759 (2001).
- [8] M.R. Mostafa, A.M. Youssef; *AFINDAD*, **7**, 121-127 (1990).
- [9] Milan Smisk; 'Active Carbon, Manufacturing, Properties and Applications', Mc-Graw Hill publisher, USA (1970).
- [10] Mohamed Mokhtar Mohamed; *Journal of Colloid and Interface Science*, **272**, 28-34 (2004).
- [11] P.K. Malik; *Dyes and Pigments*, **56**, 239-249 (2003).
- [12] Ronaldo Zanzi; 'Pyrolysis of Biomass', Royal Institution of Technology, Chemical Engineering Department, Stockholm (2001).
- [13] Steve Kvech, Erika Tull; 'Activated Carbon Processing', Civil Engineering Department, Virginia Technology Institute USA (1998).
- [14] V.P. Della, I. Kühna, D. Hotza; *Materials Letters*, **57**, 818-821 (2002).
- [15] Yupeng Guo, Kaifeng Yu, Zichen Wang, Hongding Xu; *Carbon*, **41**, 1645-1648 (2003).
- [16] Yupeng Guo, Shaofeng Yang, Kaifeng Yu, Jingzhe Zhao, Zichen Wang, Hongding Xu; *Materials Chemistry and Physics*, **74**, 320-323 (2002).
- [17] Zhonghua Hua, M.P. Srinivasan; *Microporous and Mesoporous Materials*, **43**, 267-275 (2001).