# Effect of impregnation temperature on the sorption of a cationic dye

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**Abstract**

***In this study, an important parameter in the chemical activation of sorbents was tested which is the impregnation temperature. For this, the chemical activation of cedar cone (an abundant forest waste in Khenchela forest in Algeria) has been tested. The impregnation temperature was varied from 10 to 40 °C including the ambient temperature while the concentration of the agent activator ($HNO_3$) was kipped constant at 2mole$L^{-1}$ and the rate of impregnation (IR) was equal to 20. The results show that the cedar cone (CC) has an important advantage that it's not required to treated it in specific temperature, the ambient temperature give a better performance of sorption of a cationic dye (Rhodamine B). The titrations of functional surface show clearly the increase in total of acid functions.***

***Keywords:*** *Cedar cone; Chemical activation; Impregnation temperature.*

## I. Introduction

Pollution of water is a current problem; various rivers, lakes and groundwater have been polluted. This pollution is of different origin resulting mainly from direct discharge of solid and liquid wastes from human activities and water washes into the environment. Indeed, these polluted waters may contain microbiological contaminants, metallic or organic, which in the absence of treatment can have a harmful effect on humans' animals or plants. Consequently, rigorous laws and norms were imposed by the states in order to controlled water pollution. Several techniques and methods of treatment were used to treat the polluted liquid effluents. The choice of technique is based on several criteria, the mainly criterion is the source of the effluent to be treated (the composition), the volume (flow rate), the cost of the processing operation, by-products derived from treatment and management, etc.

The techniques most often encountered are: coagulation - flocculation, ultrafiltration, reverse osmosis, biological treatment, etc.

The biological treatment showed weaknesses in the case of some non-biodegradable organics, or even toxic to microbial flora and very harmful to the proper functioning of treatment plants, while the ultrafiltration and reverse osmosis are limited in use since their very high cost.

The solution was to found a technique which provided a compromise between the cost and effectiveness of treatment, the only treatment method that addresses these two key criteria has been activated carbon adsorption. The main weakness of the process was the high cost of activated carbon and difficulties or even the impossibility of its regeneration. In this context, sorption appears to be a very interesting alternative using natural biological materials or byproduct and prepare from their activated sorbent by chemical or physical treatment.

In this study we have treated cedar cone an abundant forest sorbent chemically using nitric acid, the optimization of conditions of treatment has been published before [1]. Another important parameter which is the impregnation temperature was tested in this work. The effect of the impregnation temperature was tested on the sorption of a cationic dye Rhodamine B, the chemical structure of this dye is presented on Figure.1. The characterization by measuring the functional surface was also studies

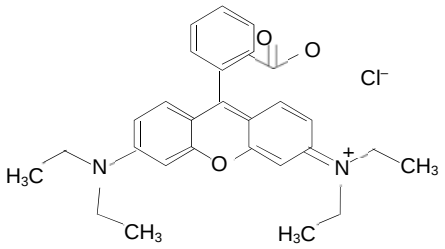


**Figure.1: Chemical structure of Rhodamine B.**

## 1. Experimental

### 1.1 Procedure of chemical activation of sorbent

Cedar cone (CC) was collected from the forest of Chelia, Khenchela, Algeria. It was washing with distilled water, oven dried at 50°C and ground and sieved to obtain a particle size range from 0.5 to 1.25 mm. After that, the grounded sorbent was washed again until the distilled wash water becomes clear. Finally, it was oven dried at 50°C. The pre-treated sorbent was chemically activated; it was mixed by nitric acid ($HNO_3$) (2mole$L^{-1}$)at impregnation ratio of 20 (volume of acid/weight of sorbent 20:1) for 24 h, then the mixture was filtered and washed repeatedly with distilled water until a yellowish color released from CC during activation became clear. Finally, the chemically treated sorbent was oven dried at 50 °C. The resulting solid product was named $ACC_N$.

### 1.2 Sorption procedure

The performance of chemical treatment of cedar cone was tested by sorption of dye in a batch technique. For this a fixed amount (2 g) of chemically treated sorbent was mixed with 200 mL of RhB solution with kipping a stirrer at 300 rpm and bath temperature at 25°C. According to the calibration curve of RhB, the $\lambda_{max}$= 554 nm. The effect of impregnation temperature was tested by varying a temperature of acid impregnation solution in the range of [10, 20, 30, 40]°C including the ambient temperature. The initial concentration of RhB solution was maintaining 50mg$L^{-1}$. The amount of sorbed dye at time ($q_t$) was calculated by the equation:

$$q_t = \frac{V(C_0 - C_t)}{m} \quad (1)$$

where $C_0$ (mg$L^{-1}$) was the initial concentration of RhB, $C_t$ (mg $L^{-1}$)was the concentration of RhB at time t, $V$(L) was the volume of the RhB solution, $m$(g) was the mass of the treated sorbent.

## 2. Functional groups

For determination of functional groups of sorbents, the technique used namely, called upon the classical Boehm's titration [2]. In this method and using spectroscopic methods and chemical reactions, has established three groups of acidic oxides area:

- ✓ G I: strong carboxylic functions,
- ✓ G II: lactones and low carboxylic groups,
- ✓ G III: phenols functions.

They are titrated according to their acid–base properties by inorganic bases or hydrochloric acid. Acid base titration was followed by a pH-meter WTW Inolab2, equipped with Electrodes SenTix® WTW high quality.

## 3. Results and discussion

### 3.1 Effect of chemical treatment of cedar cone

The treatment of cedar cone by nitric acid is represented on the Figure.2. This figure show clearly the efficiency of chemical treatment on the cedar cone, the capacity of sorption of $ACC_N$ is

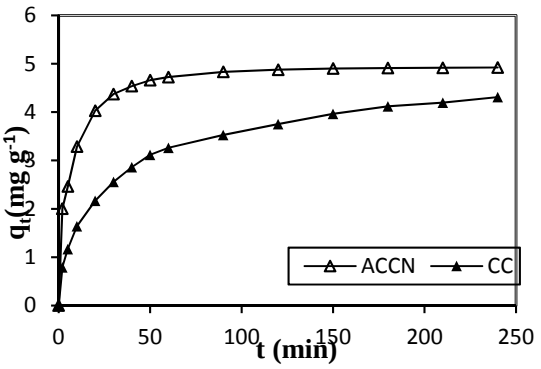


**Figure.2: Kinetic sorption of dye by $ACC_N$ and CC**

greater than CC at however time of sorption, we can observe also that the equilibrium time was reduced significantly from 300 min to 60 min. The reducing in equilibrium time and the increased in amount of dye removal can be attributed to the apparition of new sites of sorption, or created of new pores.

### 3.2 Effect of temperature impregnation

The impregnation temperature was varied between 10 and 40°C including room temperature, for this a fixed amount of Cedar cone (CC) was mixed with a known volume of nitric acid (2 mole $L^{-1}$) to keep the impregnating ration equal to (20: 1). The effect of the impregnation temperature of the sorbent was tested on the sorption of Rhodamine B. The results obtained are shown in the picture below.

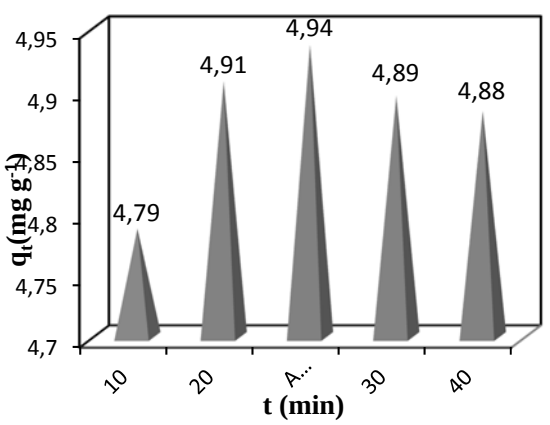


**Figure.3: Effect of temperature impregnation of Cedar Cone by nitric acid.**

As shown in this Figure the impregnation temperature has no effect on the evolution of sorption of dye, the ambient temperature seems the best temperature impregnation specially that the difference between the amount of dye sorbent is almost identical, except for the temperature of 10°C when a little decrease has been observed. This result gives to cedar cone a particular property because this parameter (effect of impregnation temperature) can be neglected during the chemical activation, view that it has no effect on the evolution of the sorption.

### 3.3 Characterization by measuring of functional surface

The results of the analysis are summarized inTable.1. These results show that during activation of CC, some groups have appeared on the surface of $ACC_N$ such as strong carboxylic acid and hydroxyl and phenol groups contrarily to the lactones and law acid carboxylic group that we have observed a small decrease compared for CC. The increase of total of acid function was expected, the treatment of sorbent by acid ($HNO_3$) has encouraged the development of new acids function and the increase in the numbers of some others. This result explains well the increase of the amount sorbed of dye by $ACC_N$ than CC, since the dye is cationic and the total of acid functions have been increased the capacity of sorption increased also.

***Table.1: Values of surface functionalities for the sorbents***

| **Group** | **$ACC_N$ ($m_{eq}\,g^{-}$)** | **CC ($m_{eq}\ g^{-1}$)** |
|---|---|---|
| Strong carboxylic acid (G I) | 0.05 | 0 |
| Lactones and law acid carboxylic (G II) | 0.09 | 0.17 |
| Hydroxyl and phenol (G III) | 0.73 | 0 |
| **Acid functions** | **0.87** | **0.17** |
| **Basic functions** | **0.02** | **0.02** |

### 4. Conclusions

Sorption of cationic dye Rhodamine B (RhB) by cedar cone (CC) (abundant forest waste) has been improved in terms of quantity sorbed or in decrease of equilibrium time after chemical activation by nitric acid (2 mole $L^{-1}$). The study of effect of impregnation temperature on the amount sorbent of treated cedar cone ($ACC_N$) shows that the ambient temperature is the best temperature impregnation. The calculated of functional surface indicates an increase in the total of acid functions of $ACC_N$ relative to the CC, this was in accordance with the results of increasing of sorption capacity of CC after treatment.

### Symbols used

| | |
|---|---|
| $ACC_N$ | Cedar Cone activated by $HNO_3$ at 2 mol $L^{-1}$ |
| $C_0$ | Initial dye concentration ($mgL^{-1}$) |
| $C_t$ | Concentration of RhB at time t ($mgL^{-1}$) |
| CC | Cedar Cone |
| IR | Impregnation ratio |
| m | mass of the treated sorbent |
| $q_t$ | Amount of dye sorbed at any time t ($mgg^{-1}$) |
| t | time (min) |
| T | Solution temperature (K, °C) |
| V | Volume of solution (L) |
| w | Sorbent weight (g) |
| $\lambda_{max}$ | wave length |

## References

[1]. Meriem Zamouche, Sihem Arris, Mossaab Bencheikh Lehocine, ' Removal of Rhodamine B from water by cedar cone: Effect of calcinations and chemical activation. Hydrogen Energy 39, 1523 -1531, 2014.

[2] Boehm H P. Chemical identification of surface groups. Advances in Catalysis 16, 179-274, 1966.