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A novel acoustic approach for the characterization of granular activated carbons used in the rum production

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ABSTRACT

Acoustic analysis and sound patterns recognition techniques have been widely used in many branches of science, however; almost none focused on the characterization of granular activated carbon. A new methodology has been developed in order to characterize activated carbon based on the dynamic analysis in audible spectra of the sound's relative amplitude power produced by water flooded on granular activated carbon. A home-build recording set-up and management of acoustic measurements have been presented and correlated with the results of porous structure of carbons characterized by N_2 adsorption. Five samples of granular activated carbons used in the rum production of different exhausted level have been evaluated by both methods. Parameters as the BET surface area and total pore volume showed a satisfactory correlation with acoustic measurement data when the signal is processed at 1000 Hz. Three frequencies components of the produced sound were analyzed and related with the porous characteristics. The found relationship gives the possibility to predict and calculate textural parameters of granular activated carbons applying the acoustic technique. This methodology approach opens possibilities in using acoustic experiments for the characterization of high-porosity materials and to determine their exhausted level.

1. Introduction

Activated carbon (AC) adsorption is the most common technique for removing various pollutants due to its extended surface area, high pore volume, well developed porous structure and specific surface functional groups [1,2]. AC can be used powdered or granular (0,2–5 mm). Granular activated carbons (GAC) are widely employed for product purification (such as sugar refining, food processing and water treatment) [2]. In spirits and liquor production as rum industry, GACs are used to remove organic compounds that affect the sensorial quality of the final product [3]. Rums are a complex mixture of organic substances: 186 organic compounds have been identified [4,5]. When GACs become exhausted in the rum production they are landfilled and replaced by fresh GACs. However, the landfilled GACs create a solid waste problem. For this reason a regeneration process should be applied and the effectiveness of GAC regeneration must be guaranteed. In order to determine the exhaustion level of GAC or the regeneration degree reached, a proper and fast analytical technique based on determination of specific surface area and porosity has to be applied [6]. However, the technological facilities of rum producers are limited. Additional this quality parameter needs to be checked on a very regular base during the whole rum production process. At present a specialized rum taster expert determines when GACs need to be replaced. A quick action needs to be undertaken from the moment the taste of the produced rum is not within the accepted high quality level. Therefore an alternative and as quick and trustable way to measure the exhaustion level of GACs is more than welcome.

This paper describes such an approach based on flooding a GAC sample with water. This results in a sound production, which can be analyzed by a proper acoustic technique. Recorded data can be correlated with GAC properties and more specific in the determination of its exhaustion level.

The GAC sound is produced by bubbles escaping from the AC cracks and pores when water molecules occupy the air filled spaces

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Nomenclature

A_1	constant parameter in data fitting
k	number of RMS samples processed per step of time's interval Δt.
L_0	average micropore width (nm)
n	discreet time
nf	discreet final time
p_0	acoustic pressure of reference (Pa) (constant)
p_a	acoustic pressure (Pa)
$\overline{\text{RMS}}(r)$	average of RMS power of reference (dB)
$\overline{\text{RMS}}(n)$	average of RMS power at discreet time "n" (dB)
RMS(k)	relative amplitude power of the signal at "k" instant (dB)
RMS	relative amplitude power of the signal (dB)
RMSF(n)	relative amplitude amplified function
r	distance from the bubble center (m)
SL	silence line
SS	sound surface
S_{BET}	specific surface area (m^2/g)
t_0	initial time (s)
t_1	constant parameter in data fitting
t_n	time at "n" discreet step (s)
V	volume of the bubble (m^3)
V_{DR}	micropore volume (cm^3/g)
V_{mes}	mesopore volume (cm^3/g)
V_T	total pore volume (cm^3/g)
X	gradient of sound power between receiver and emitter
x	amplification factor
ρ_L	density of the liquid (kg/m^3)
$\Delta t'$	time interval of sampling to RMS values (mls)
Δt	step of time's interval (s)

inside of the GAC by displacing the present air. The amount and size of produced bubbles are closely related to the porosity of the GAC. The bubble size, the volume fraction and rate at which the bubbles appear by approaching the water surface influence sound parameters as frequency and amplitude [2,7–13]. In the case of GAC used in the rum production, large amounts of organic taste compounds with different molecular sizes adsorb, and block cracks and pores of GAC, creating an important reduction of pore volume and specific surface area. Exhausted GACs therefore result in a reduction of bubbling potential and consequently in a reduction and a change in the sound level.

The use of acoustic measurements makes it possible to determine the overall porosity but also to characterize the porous structure of GAC according to the sound patterns obtained. Bubbles and its corresponding sound patterns are in general widely studied, theoretically analyzed and applied in many scientific fields [14–17]. The analysis of bubble sound patterns to characterize mechanical events such as pump cavitation or cavity effects in gas-jet impingement or propellers have proven to be accurate [18,19]. Due to the complexity involved in bubble acoustic and mechanical vibration modeling, a previous knowledge about signal processing, math and physics is needed to face a deep interpretation of obtained results. This work is a first approach to use acoustical techniques to study porous characteristics in high-porosity materials.

2. Materials and methods

2.1. GAC samples

Five samples of GAC (0.8 mm) were obtained from the major rum producer in Cuba: labeled as AC-1, AC-2, AC-3, AC-4 and AC-5. The samples AC-1 is a fresh GAC (virgin) and AC-5 is the most exhausted GAC used in the rum production process. The others have different exhaustion levels ranging between them.

2.2. Samples characterization

The porous structure of GACs was also characterized by N_2 adsorption at 77 K using ASAP2020 (Micromeritics). Before the analysis, the sample was degassed overnight at 300 °C. The specific surface area (S_{BET}) was estimated by the BET equation. The amount of nitrogen adsorbed at the relative pressure of $p/p_0 = 0.96$ was employed to determine the total volume of pores (V_T). The micropore volume (V_{DR}) was calculated by applying the Dubinin-Radushkevich equation. The difference between V_T and V_{DR} was taken as the mesopore volume (V_{mes}). The average micropore width L_0 was calculated using the Stoeckli equation [20]. The quenched solid density functional theory (QSDFT) was used to determine the pore size distribution [21].

To observe the morphology of GAC grains, a scanning electron microscope (SEM) (Vega®Tescan/TS5130SB/SE Detector) was used.

2.3. Preparation of GAC samples for sound experiments

The GAC samples were dried at 200 °C during 1 h using a Boxun oven: (BGZ Series). Samples were held overnight in a silica-gel dryer. Five samples of each GAC of 10 g were weighed, labeled and kept in sealed envelopes.

2.4. Sound experiment on GAC samples

2.4.1. Experimental set-up [22]

The GAC bubble sound experiments were performed in a home-made sound enclosure box (Fig. 1). The sound enclosure box was built using an external coffer of soft wood which was internally covered with two layers of soft foam and sponge (one inch thick) to absorb not only possible external interferences but also the unwanted resonant vibration produced by the vessel during the measurement process.

Internal parts of the sound enclosure box consist of:

1. Microphone (Model:Samsom R21/unidirectional cardioid polar pattern/frequency range 20–20,000 Hz/ sampling rate 44,100 Hz).
2. Erlenmeyer flask (250 ml).
3. Coaxial cable for connecting microphone, amplifier and PC.
4. Injection tube (diameter: 3 mm).
5. Separator funnel (100 ml).
6. Valve.

2.4.2. Working description [22]

The sample of GAC (10 g) is put into the Erlenmeyer flask (2) (Fig. 1) and distributed uniformly on the bottom. The injection tube (4) is introduced inside of the Erlenmeyer flask and the microphone (1) is adjusted capping the Erlenmeyer flask using a special foam-sponge gasket avoiding the rigid interconnections between the microphone and the vessel reducing the possibility