The Effect of Integration Between Heat and Time Factors on the Efficiency of the Carbonization of Peanut Shells to Prepare Biochar

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ABSTRACT:

This research work was carried out in the soil fertility laboratory and plant nutrition at Faculty of Agriculture-Tishreen University-Lattakia, summer season 2019, by studying the effect of integration between heat and residence time factors on the efficiency of the carbonization of peanut shells to prepare biochar. The experiment included (12) treatments, consisting of (4) levels of carbonization heat (350, 450, 550 and 650 C^o) in combination with(3) levels of carbonization residence time (15, 30 and 60 minutes). After the completion of the carbonization process according to the specified temperature and time, the carbonized samples were cooled and preserved to do some measurements and tests. Some tests were performed to state the heat and time that gives the best efficiency of carbonization process, by color comparison, percentage of organic carbon, biochar purity and the percentage of ash. The results of the color changes of the carbonization products and alcohol leachates, match to the results of the chemical organic carbon estimation, where the percentage of organic carbon in the carbonization products recorded the highest value at a temperature of (350 C^o) which is (2.05 %) and recorded the lowest value at a temperature of (650 C^o) which is (1.2 %). There was an inverse clear significant relationship between the concentration of organic carbon and both the carbonization temperature and the carbonization residence time. Also the good biochar with high percentage of nonorganic carbon(about 79-80 %) and little or no Or.C, was obtained by carbonization at heating temperature of 450 C^o for 1 hour or longer duration, or at higher heating temperatures (550 and 650 C^o) for shorter durations.

Keywords: *Biochar, Carbonization, Peanut shells, organic carbon.*

I. INTRODUCTION

Biochar currently has a special research attention after its importance has been confirmed in agricultural and environmental applications as it is a porous carbon material produced by the pyrolysis of the biomass under limited or no oxygen presence, as its particles form very high specific surfaces with high functional groups of negative and positive charges. These properties prompted researchers to pay attention to the applications of this product in improving the chemical and water properties of the soil, as well as its importance in carbon sequestration for thousands of years. Which reflects positively on the environment and contributes to mitigating the negative effects of climate changes, especially as industrial plants produce huge quantities of carbon dioxide emissions into the atmosphere, and agricultural lands in general, face the problem of nitrogen leaching as a result of excessive use of nitrogen fertilizers, which threatens the quality of underground water and causes the phenomenon of nutritional enrichment. The use of biochar to improve soil properties goes back to the black amazon lands known as, Terra Preta de Indio, in the Amazon basin, where it showed that seasonal additions of the charred crop residues to soils, contributed to improving their physical and chemical properties and sustaining their fertility [1].

After being universally convinced of the importance of Biochar at the agricultural and environmental levels, the technology of carbonization began to reveal a very promising future, and the need to characterize and define the characteristics of the biochar produced from any biomass under different conditions of carbonization to understand the properties most relevant to its use as a soil amendment and most affected by charring conditions. In spite of the long research about the carbonization process, more technical research is needed to improve the quality and efficiency of the carbonization process with minimum loss of biomass and maximum yield of good biochar.

As the biochar particles are not homogeneous, the aromatic structures and chemical composition of the molecules make it difficult to get similar single particles of biochar [2]. Therefore it is necessary to understand how the quality of biochar is affected by the appropriate selection of biomass and the carbonization conditions [3].

Biochar is produced globally at the investment level for agricultural uses, using advanced carbonization systems [4]. These systems allow monitoring the process conditions in advance, through which the physical and chemical properties of biochar produced from such carbonization process can be influenced. However, traditional biochar production technologies are still in use [5], and developing these technologies requires pursuing research in this area.

Biochar particles contain microscopic crystalline molecules consisting of a large number of mineral carbon atoms that form the aromatic rings, which are compounds resided by hexagonal rings of carbon atoms linked in form of longitudinal sheets or layers forming what is called biochar or (graphite) [6], with some solid impurities and spaces which in turn form large and small pores between layers and give it a porous bulk.

Biomass properties, along with the carbonization temperature and the time of heat, are the main factors affecting biochar production and its specific surface formation. The availability of nutrients in biochar is also associated with differences in the biomass composition involved in the carbonization process [7], the moisture content [8] and the thermal conditions of the same process [9], because these factors control the degree of formation of aromatic rings, and the fixing of carbon atoms in the biochar structure, where at high temperatures the carbon atoms are distributed in multiple and dense aromatic structures [10]. These aromatic structures have low O/C ratio making it more resistant to microbial degradation, ([11], [12]) which is very important property for biochar to resists microbial degradation and to be best suited for carbon fixation for thousands of years. Re. [1] mentioned that the pyrolysis process of the biomass during the carbonization process, is complicated due to the large variation between the components of its various sources and the diversity of potential thermo chemical reactions, but slow pyrolysis is the traditional method to prepare biochar. Generally, pyrolysis breaks down the polymeric chain of the complex structure of the biomass (cellulose, hemicellulose, lignin, fat ....), to produce a group of solid products consisting mainly of carbon atoms in the form of crystalline molecules (graphite), in addition to liquid and gas compounds that evaporate or volatilize outside the charred mass leaving what is called biochar with some solid impurities of calcium carbonate and mineral elements, in the presence of little or no oxygen, as the carbonization heat plays a major role in the development of the biochar tissue structure by rearranging the crystalline structure of the solid components [13].

During the pyrolysis process, the biomass components of cellulose, hemicellulose, and other compounds are decomposed thermally and produce intense organic liquids such as tar, furfural, acetic acid, acetone, methanol, water vapor and non-condensable gases, while lignin structures decompose to give biochar [14].

The biomass productivity of biochar, depends on the quality of the raw materials and the conditions of carbonization such as heating temperature and period of heating, in which the biochar is formed, where at high temperatures of carbonization, woody pieces are usually more carbon rich compared to other biomaterials such as sewage sludge and animal remains [1]. Therefore, woody biomasses were considered the most important sources of biochar production according to [15], because they give the highest percentage of biochar compared to other types, because they contain large quantities of cellulose, hemicellulose, lignin and small amounts of organic and inorganic extracts [16].

In a new study, the carbonization process for biochar preparation from the residues of turnip stems in China, [16], found that the carbonization heating temperature of the biomass of these residues, directly affected the amount of biochar produced and the carbon fixation ratio. And that the increase in the temperature of carbonization from (200 to 700 C^0) increased the percentage of mineral carbon from (47.5% to 80.2%), and the percentage of accompanying impurities like hydrogen, decreased from (5.9 to 1.3%) and oxygen from (39.5 to 5.3%). Re. [17], also, found that the biochar content of the accompanying impurities of nitrogen, hydrogen and oxygen decreases with an increase in the carbonization temperature from (400 to 700 C^0), while its non-organic carbon content followed the opposite trend with increasing temperature.

Also, the same researchers found similar results on biochar prepared from the residues of apple tree clippings, where the results indicated that the high temperature of carbonization increases the content of non-organic carbon and some minerals (K, P, Fe, Zn, Ca, Mg). In the same context, re. [18] found that raising the carbonization temperature up to (500 C^0) produces biochar more rich in nutrients (P, K, Ca. Mg), with non-organic carbon content of (78%).

In the field of preparing good quality biochar for agricultural use, many researchers focused on some of the important characteristics that are affected by changes in the temperature of pyrolysis as the percentage of non-organic carbon and the need to get rid of some toxic impurities of plant roots such as tar and volatile oils formed during the pyrolysis process, because of its negative effect on biochar properties and soil biological activities. In this regard, some researchers pointed out that the low temperature of the carbonization process leaves more toxic effects to the plant due to the persistence of some tar and other toxic organic compounds in biochar [19].

Re. [10] indicated that with the rise of pyrolysis temperature of the biomass of rapeseed trunks from (200 to 700 C0), the non-organic carbon content in the biochar increased from (13.3 to 75.2%), but tar decreased by (81.8 to 9%), while phenol and carboxyl

acidic groups were decomposed and volatilized by increasing the temperatures of carbonization. The effect of the heating residence time factor was less, as the ash content increased during the carbonization process from 8 to 10.2%, with heating time advancing from 10 to 100 minutes at the same temperature, due to the availability of time required for the release of mineral elements from organic complexes and their conversion to metal oxides and salts.

Re. [20] found that the slow pyrolysis of biomass during the carbonization process gives more biochar. Also, re. [21] found that increasing the carbonization temperature of the biomass of sorghum cones from (400 to 600 C^0) increased the percentage of non-organic carbon from (73.2 to 82.2%) in the resulting biochar. As the biomass carbonization temperature increases, the biochar and ash content tend to increase while non-carbon impurities such as hydrogen, oxygen, nitrogen, H/C and O/C ratios tend to decrease, according to re. [22], the same was found with the biomass of poplar wood by [23].

Re. [24] observed that biochar produced by high temperature carbonization of the biomass has a greater potential to stabilize carbon more resistant to degradation when added to soil compared to biochar produced by low temperature. In another study by re. [25] they found that the biochar produced by the carbonization process at high temperatures contains a greater number of un-degradable carbon particles, and they attributed this phenomenon to the formation of aromatic structures, where the oxygen and hydrogen are released from the biochar array, making it more aromatic, and therefore more resistant to decomposition. This study also showed that the resistance of biochar to decomposition is not only related to the temperature and time of carbonization, but also to the source of the biomass involved in the process of carbonization, where a difference in the decomposition resistance of biochar resulting from several sources of biomass at the same temperature is observed.

II. MATERIALS AND METHODS:

A. The raw material of biomass:

In this study, the raw materialof peanut shells (crusts) was used as a biomass for carbonization to prepare biochar.

B. Experimental design and implementation:

The experiment included (12) treatments, consisting of (4) levels of carbonization heat (C) (350, 450, 550 and 650 C^0) in combination with (3) levels of carbonization residence time (15, 30 and 60 minutes), as shown in Table (1):

Table (1): shows number of samples and treatments codes

Treatment number	1	2	3	4	5	6	7	8	9	10	11	12
Carbonizationheat – C (C^o)	C_1 = 350 C^o			C_2 = 450 C^o			C_3 = 550 C^o			C_4 = 650 C^o		
Residence time–T (minutes)	T_1	T_2	T_3	T_1	T_2	T_3	T_1	T_2	T_3	T_1	T_2	T_3
	15	30	60	15	30	60	15	30	60	15	30	60

The experiment was carried out in the soil fertility laboratory and plant nutrition, in soil and water Science Department, Where homogeneous samples of peanut shells weighing (50 g) per sample were placed in 100 cm^3metal cylinders, covered with heat-resistant aluminum foil to prevent the entry of oxygen, perforated to exit of combustion gases and water vapor, (Figure 1).

Figure (1): Peanut shells samples in metal cylinders intended for carbonization.

The used cylinders were placed in the furnace at stated temperatures with different carbonization periods according to the above-mentioned treatments. After the completion of the carbonization process the samples were taken out and placed to cool down. Then, the resulting weights of the residues, were recorded and the loss of biomass by carbonization was calculated for treatments and attributed to the original weight as a percentage of the biomass before the carbonization. After that the carbonized samples were preserved in small plastic containers, well stoppered, marked with treatments numbers, temperature and time of charring, and kept for lab measurements and tests.

C. Measurements and Tests:

1) Percentage of Biochar and Loss by Carbonization:

The percentage of loss of biomass for peanut shells as a result of the carbonization process was determined by applying the following equation:

$$\%\text{loss by Carbonization} = \frac{B1 - B2}{B1} \times 100$$

whereas:

B1 = weight of biomass sample for peanut shells before carbonization.

B2 = Weight of biomass sample for peanut shells after carbonization.

Accordingly, the percentage of Biochar was determined as follows:

% Biochar = 100 – % loss by Carbonization

After the completion of the carbonization process, some color, physical and chemical tests were performed to determine the efficiency and degree of completeness of the carbonization process and the biochar produced as follows:

2) Organic Carbon Test:

One of the evidence of incomplete carbonization was the presence of organic carbon in the carbonization remains. It is done by washing a small samples with ethyl alcohol and observing the presence of the brownish color in the leachates. Where the procedures of washing samples of equal weight (1 g) of the residues of the carbonization process were carried out for each treatment with equal quantities of ethyl alcohol (50 ml) followed by (50 ml) of distilled water, and the filtrates were collected in conical flasks of 100 ml, then the color differences between treatments were documented as a pictures supported with the chemical analysis for organic carbon in the samples as an evidence of its presence beside the biochar in the residues of the carbonization, since the degree of its presence in the leachates has a reverse relation with the degree of completion of the carbonization process.

3) Chemical Determination of Organic Carbon:

The presence of the organic carbon in the biochar was estimated by the color method according to re.[26], after wet digestion with potassium dichromate method according to [27], and the results of the analyses were combined with the color pictures of the leachates.

4) Degree of Purity of Biochar and Percentage of Ash:

The degree of biochar purity and ash percentage was determined by burning biochar samples weighing (X1= 5 g) each, in a porcelain crucibles at a temperature of (500 C^o) for (5) hours, cooled down, and the resulting ashes which represent the mineral impurities were weighed (X2). Then the purity of the biochar and the percentage of ash in each sample were calculated as follows:

$$\%\ \text{Biochar} = \frac{X1 - X2}{X1} \times 100$$

$$\%\ \text{Ash} = 100 - \%\ \text{biochar}$$

5) Statistical Analysis:

The results of the experiment were subjected to analysis of variance (Two ways ANOVA). Also, the least significant difference (LSD), was calculated, at 5% level of significance, for the mean values of the treatment variables and their interactions, using the statistical program Genstat 12th Edition [28], as shown in table 2.

III. RESULTS AND DISCUSSION

A. The Efficiency of the Carbonization Process:

The efficiency of the biomass carbonization process could be well-founded and defined through the decrease of the percentage of impurities and the increase of the percentage of biochar in the residues after carbonization. Accordingly, the appropriate temperature and duration for complete carbonization process which assure the convert of the organ metallic compounds to oxides and mineral salts, and getting rid of oxygen, hydrogen, water vapor and some volatile elements and finally transform organic carbon to nonorganic carbon called biochar or charcoal (graphite) at the highest possible rate. This was determined by the following tests:

B. The Color comparison:

Direct color pictures were taken for the residues after carbonization process of the biomass for all treatments according to the temperatures and period of time of this process. As the color of the residues changes from light brown to dark brown and black, the more efficient and more complete the carbonization process.

Figure (3) also shows the color changes of the leachates of washed product samples with ethyl alcohol from dark to light brown or no color and the percentage of organic carbon in them under the effect

of heating temperatures between (350C° - 650C°) over (3) residence times of the carbonization process. Where alcohol has been used for a long time to dissolve and extract some highly complex organic compounds from organic matter such as fats, waxes, resins and lignin by many researchers [29] and [30].

Figure (2): shells samples on color changes of the products.

Where the brown color in the leachates indicates the presence of some organic carbon that has not converted to biochar, and vice versa the clear color of the leachates indicates a low or absence of organic carbon, whose presence in the (biochar) is considered as an impurity, and indicates that the carbonization process is incomplete. The color changes of the carbonization products and the alcohol washings leachates counterpart with the content of the organic carbon, as the percentage of organic carbon in the biochar products recorded the highest value at a carbonization temperature of (350 C°) which is (2.05 %) and recorded the lowest value at a temperature of (650 C°) which is (1.2 %). There was a clear inverse relationship between the organic carbon concentration in the produced biochar and both the heat and period of carbonization, figure (3). This result agreed with the findings of [16], [10], [17], where they mentioned that the biochar content of organic carbon decreased and the percentage of non-organic carbon increased with increasing carbonization temperature.

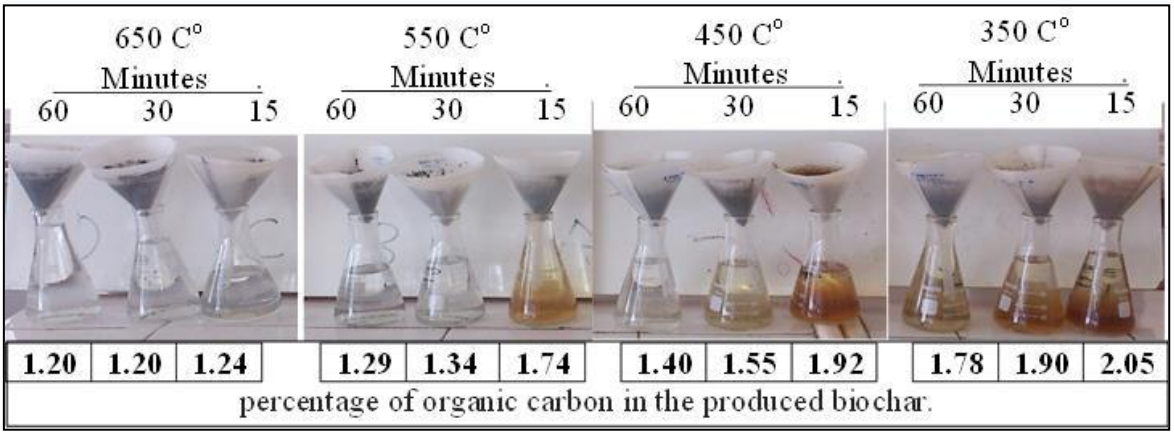

Figure 3: the effect of different temperatures and duration of carbonization of peanut shells samples on color changes in the leachates of washed produced biochar samples with alcohol and the percentage of organic carbon in them.

C. Biochar purity and the impurity ratio:

Table (2) shows the Average weight values of the loss in biomass of peanut shells after carbonization under the effects of (4) levels of carbonization heat in combination with (3) residence time, as well as the degree of purity of the biochar in the carbonization products. The table clearly shows that there are highly significant effects (F= <0.001) for the combinations between the heat and the carbonization residence time factors on the residues percentage of the biomass after carbonization, Where these values decrease with the increase in heating temperature of the carbonization from 350 C^0 up to about 650 C^0 passing through 450 C^0 and 550 C^0 and with increasing the carbonization residence time from (¼) to (½) and then to (1) an hour. The carbonization process started to complete and end after the temperature reaches (450 C^0) for one hour or (550 C^0) for (½)an hour or (650 C^0) for (¼) an hour or longer, noting that the carbonization process did not complete and reach the stability phase at (350 C^0) if the carbonization residence time was less than an hour, or at (450 C^0) if the carbonization residence time is less than (½) an hour. These results were comparable with the results of [22] and [13] how observed a decrease in the residues percentage of biomass with increasing carbonization temperature. The net return of the biomass is expressed as the difference between the weight of the biomass before carbonization and the loss by ignition during the carbonization, it was high (51.7–81.16%) and associated with a low percentage of biochar in the carbonization products (54.2–58.2%), when the carbonation process was not complete at (350 C^0) for one hour or (450 C^0) for (½) an hour or (550 C^0) for (¼) an hour, while the best percentage for net return of biomass ranged between (42.92 %) at (450 C^0) for one hour and (41.3 %) at (650 C^0) for a residence time of (¼) an hour which achieves the highest percentage of biochar (79–80%) in the carbonization output according to Table (2).

Table (2): Effect of temperature and residence time of carbonization on the average percentage of the loss in biomass of peanut shells through the carbonization process, along with biochar impurities.

biochar %	impurities %	% Weight after ignition	% Loss in Ignition	residence time minutes	temperature C^0
54.20	45.80	81.16	18.84	15	350
55.40	44.60	74.1	25.9	30	
56.60	43.40	51.7	48.3	60	
57.00	43.00	77.02	22.98	15	450
58.20	41.80	51.7	48.26	30	
79.00	21.00	42.92	57.08	60	
57.80	42.20	51.12	48.88	15	550
79.20	20.80	41.94	58.06	30	
80.00	20.00	41.88	58.12	60	
80.00	20.00	41.3	58.7	15	650
79.60	20.40	41.42	58.58	30	
79.60	20.40	41.36	58.64	60	
<.001	<.001	<.001	<.001	-	F.pro
0.515	0.515	1.659	1.659	-	$LSD_{0.05\%}$

The low percentage of biochar and the high percentage of impurities in the product, indicate a low efficiency and incomplete carbonization process, While the rise of biochar percentage at its highest level (79.2-80%) along with a minimum impurities (20-20%), mean high efficiency of the carbonization process. These results were consistent with the results of [31] and [14] where high percentage of impurities and low biochar are evidence of the inefficiency of the carbonization process.

IV. CONCLUSION

1. the appropriate temperature and residence time for the biomass carbonization process could be decided and approved by observing and measuring some physical and chemical characteristics for the produced biochar after the carbonization process, such as; Color, Or.C, % of biochar and % of impurities.
2. The suitable temperature for the proper carbonization varied between (450 C^o) for one hour, (550 C^o) for (½) an hour and (650 C^o) for (¼) an hour or longer durations to get high efficiency of the biomass carbonization process with average net return about (41 %) of the biomass as biochar, while lower temperatures need longer time to complete the carbonization process with no or less impurities.
3. Good biochar with high percentage of nono-rganic carbon (about 79-80 %) and little or no Or.C , could be obtained by carbonization temperature of (450 C^o) for one hour or longer residence time, or at higher heating temperatures for shorter residence times.

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