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Production of activated carbon from cocoa pods: Investigating benefits and environmental impacts through analytical chemistry techniques and life cycle assessment

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ABSTRACT

Activated carbons currently represent a feasible adsorbent substrate for the removal of organic and inorganic compounds from solutions, due to their large specific surface area and high porosity. The chemical characterization of activated carbon and the environmental burden related to its production are a crucial point that challenges researchers all over the world for the generation of feasible absorbents at low cost, with low environmental impacts. This work aims at evaluating the preparation of activated carbons from cocoa pods using the Response Surface Methodology (RSM), and Life Cycle Assessment (LCA) to evaluate the environmental impacts of the process. Analysis of variance (ANOVA) and *t*-test are used to determine the major contributing factors to the production process. Results show that at a calcination time and temperature of 3 h and 600 °C respectively, coupled with an amount of 0.6 mol/L of potassium hydroxide (KOH) as activating agent are needed for the most suitable activated carbon production, in terms of maximum iodine number (995 mg/g) and yield (74.4%). The raw biomass and the produced activated carbon are characterized by thermogravimetric analysis (TGA), scanning electron microscopy (SEM), X-ray diffraction (XRD) and infrared spectroscopy (FTIR). The quantification of the major environmental impacts generated during the production process of activated carbon are analyzed through Life Cycle Assessment. Results show that the major contributor to environmental impact is the electricity used in the laboratory steps, with an average contribution throughout all the impact categories of almost 70%, a minimum to land use potential (≈9%), and a maximum to freshwater eco-toxicity (≈99%). Toxicity is related almost exclusively to the electric energy used (average ≈ 93% of contribution). Electricity is also the major contributor to Freshwater eutrophication potential (≈70%) together with distilled water (≈20%).

1. Introduction

Activated carbons have become today the most suitable materials for water purification processes, discoloration and biogas purification, due to their relatively low cost, high porosity and high surface area (Arena et al., 2016). They are most often obtained from carbon rich substrates, namely lignocellulosic materials, agricultural by-products and fossil or biogenic residues (Jawaduddin et al.,

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List of abbreviations

AC	Activated Carbon
RSM	Response Surface Methodology
LCA	Life Cycle Assessment
ANOVA	Analysis of variance
TGA	Thermogravimetric analysis
SEM	Scanning electron microscopy
XRD	X-ray diffraction
IR	Impregnation ratio
TS	Total solid
VS	Volatile solid
LCI	Life Cycle Inventory
DS	Differential scanning calorimetry
PMFP	Particulate matter formation potential
FETP	Freshwater eco-toxicity potential
FEP	Freshwater eutrophication potential
GWP	Global warming potential
HCTP	Human carcinogenic toxicity potential
HNTP	Human non-carcinogenic toxicity potential
IRP	Ionizing radiation potential
LUP	Land use potential
METP	Marine eco-toxicity potential
MEP	Marine eutrophication potential
MSP	Mineral resource scarcity potential
OFHP	Ozone formation, Human health potential
OFTP	Ozone formation, Terrestrial ecosystems potential
TAP	Terrestrial acidification potential
TETP	Terrestrial eco-toxicity potential
WCP	Water consumption potential

2019).

Currently, more and more research is being carried out on the production of activated carbon using agricultural by-products thanks to their wide availability, abundance and especially their low market cost. Furthermore, the conversion of agricultural by-products into activated carbon could theoretically contribute in preserving the environment by reducing the emissions generated by landfilling and/or incineration of organic materials (Jhatial et al., 2018).

Cameroon is the world's fourth largest cocoa producer, with currently more than 600,000 cocoa farmers. It can be estimated that more than a million people, one in five Cameroonians, are directly interested in its farming. In other words, the whole economic life of the country, the very future of its development, is linked to the fate of cocoa. Cameroon produced about 264,253 tonnes of cocoa in 2019; with an increase of 4.2% from the previous season. The main finished products obtained after processing cocoa fruits are butter and chocolate. Though, significant quantities of cocoa pods are generated during cocoa processing.Cocoa pods represent approximately 75% of the total weight of cocoa fruits (Cruz et al., 2012). This waste is most often burnt in open air, thus generating significant environmental degradation. The use of cocoa pods as raw material to produce more valuable products is a very attractive venture ecologically and economically (Rufis et al., 2019). Activated carbons are generated from agricultural by-products through a carbonization process followed by chemical or physical activation (Jawaduddin et al., 2019). Producing activated carbons by chemical activation with potassium hydroxide yields products with high specific surface and micro porosity. The production of activated carbon from cocoa pods provides an economical means for a profitable product, at the same time contributing in solving the issue of agricultural waste management.

Several researchers have developed much interest in using agricultural by-products as raw materials for producing adsorbents at reasonable costs (Senthilkumar et al., 2017), as activated sludge in membrane bioreactors., very effective in the treatment of wastewater, even if presenting limitations related to their operations (Arsalan and Mohammad-Hossein, 2018).

Activated carbon is regularly used in many areas of daily life. Since these activated carbons have a high specific surface area and porosity, they have a high capacity to eliminate organic pollutants by adsorption (Koçer and Acemioğlu, 2016). Several activating agents (NaOH, H_2SO_4, H_3PO_4 etc.) can be used to activate the biomass. Cruz et al., 2012 prepared activated carbons from cocoa pods by chemical activation with KOH by varying the calcination temperatures (Koçer and Acemioğlu, 2016). This production technique has limitations in that it does not take into account the interactions between the various parameters studied.

The production of good quality activated carbon requires a perfect mastery of the various stages of the synthesis process and the factors that influence this preparation. The traditional method of studying the effect of certain factors on production process is performed by keeping all the factors fixed at a certain level, except one which is varied by focusing on determining the related optimum condition. This is then repeated for all the other parameters. The disadvantages of this method are: (i), the inability to determine the interactive effects between these factors, and (ii) the need to carry out a very large number of experiments which consequently require much experimental time (Alam et al., 2007). This is why the surface response methodology is used to determine the significant factors and the correlation of these different factors with a desired response. Optimization by the experimental design method makes it possible to simultaneously study several factors involved in the synthesis of activated carbon.

Although the production of activated carbon from cocoa pods is environmentally friendly and economical, previous works suggest that a full estimate of the environmental impacts ranging from the cultivation of the pods to their transformation into activated carbon has not yet been established. Available scientific literature only shows a few works carried out in the assessment of the environmental impacts associated with the production of activated carbon (Arena et al., 2016). However, the environmental impacts assessment associated with a specific activated carbon shows the relation with the raw material, as well as the pretreatment, carbonization and activation steps. To date, no in-depth study coupled with the production of activated carbon at laboratory scale followed directly by its environmental impact assessment has yet been carried out.

The current context marked by the general awareness of the importance of protecting the environment and the impacts caused by production processes, greatly justify the interest shown toward potential impacts and environmental costs arising from activated carbon generation. Life cycle assessment (LCA) aims to investigate environmental impacts of human activities in order to comply several requirements: quality and performance, technical feasibility, cost control and respect for the environment (ISO 14044, 2006). The analysis thus plans to identify viable solutions in order to increase the environmental performance of the production system, while being part of a global framework oriented towards the development of an environmental management system. The associated question raised in this work concerns the environmental aspect of the process. How to quantitatively determine the environmental impacts of activated carbon generation from cocoa

Fig. 1. Waste cocoa pods.

pods? The objective of this work is to optimize the main factors that influence the preparation of activated carbon from cocoa pods using RSM and to assess the potential environmental impacts of the conversion of the cocoa pods to activated carbons in Cameroon. The main interest of the present study is to fill the lack of knowledge about the environmental performance of the production of activated carbon from cocoa pods.

This study is organized in three stages: the first step aims at optimizing the main parameters that influence the preparation of activated carbon. The second step focuses on the different physicochemical characterizations of the best activated carbons. Finally, the third step highlights the evaluation of the potential environmental impacts of the investigated system.

2. Materials and methods

2.1. Materials

The biomass used in this work is cocoa pods (Fig. 1). Cocoa pods are converted to activated carbons by chemical activation using potassium hydroxide as activating agent. The biomass is collected in the locality of Menoua in West Region, Cameroon. All the chemicals used are of analytical reagent grade. The choice of KOH in this study as activating agent is justified by the production of activated carbons at low calcination temperatures with a very large pore structure and a considerable specific surface.

2.2. Methods

2.2.1. Pre-treatment

The raw material is washed with pure water to remove any dust and then dried at 105 °C, for 24 h, to remove moisture. The dried biomass is then ground and sieved to a size range of 1–2 mm. Subsequently, 10 g of the sieved particles are selected and mixed with KOH pellets, using a 1:10 impregnation ratio (IR), where 1 and 10 respectively represent the mass of activating agent and of raw material. The IR was estimated from equation:

$$IR = \frac{\boldsymbol{mass\ of\ KOH}}{\boldsymbol{mass\ of raw\ material}} \quad (1)$$

The impregnated biomass is dried at room temperature overnight and then at 105 °C for 1 day using an oven. The dried material placed in a ceramic crucible is then calcinated at a heating rate of 5°/min using a furnace. Concentration of activating agent, calcination time and calcination temperature are set according to RSM. After the calcination process and cooling of the furnace, the samples obtained are washed with a 1.0 mol/L HCl solution and then with distilled water. The various activated carbons thus obtained are dried for 24 h at 120 °C before being crushed to obtain powdered activated carbon.

2.2.2. Experimental design

Box-Behnken designs, Doehlert designs and centered composite designs are the most commonly used response surface designs (Goupy et al., 2006). Composite designs allows starting the study with a minimum number of experiments. If the model is validated, then the study usually ends. Otherwise, more experiments can be performed without losing the results of previous tests. Response Surface Methodology, with Central composite design (CCD) is used to optimize the parameters for preparing the activated carbons. The independent variables chosen are the carbonization time, temperature and activating agent concentration to perform 17 experiments with repetitions using CCD with 3 points in the centre. Each test is carried out three times with the aim of minimizing the errors. The variables X_i, X_j and X_z respectively are chosen to represent the quantitative factors which influence AC preparation. The field of study is $450 \leq X_i \leq 600$ °C; $0.3 \leq X_j \leq 0.6$ mol/L; and $1 \leq X_z \leq 3$ h. The response values (Y) are the iodine number (Y_j, mg/g) and the yield ($Y_{i,\ \%}$) of the activated carbon. The different responses are analyzed by means of a developed model correlating the response to the three different variables using a second order polynomial given by equation (Sibiescu and Cretescu, 2015):

$$\boldsymbol{Y = I + aX_1 + bX_2 + cX_3 + dX_1^2 + eX_1X_2 + fX_1X_3 + gX_2^2 + hX_2X_3 + iX_3^2 + \varepsilon} \quad (2)$$

where: Y is the predicted response, I is the constant coefficient; a, b and c the linear coefficients, d, g and i the quadratic coefficients, e, f and h the interaction coefficients, X_1, X_2, X_3, X_1^2, X_1X_2, X_1 X_3, X_2^2, X_2X_3, X_3^2 are the coded values of the variables considered and ε represents the error.

2.2.3. Analytical methods

Some chemical analyses are performed in order to determine and quantify the nutrients of the different substrates. The qualitative and quantitative analyses of the macromolecules (K, Pb, Zr, Cu, Fe) present in the different substrates are carried out at the Laboratory of Chemical Engineering of the Faculty of Industrial Chemistry of the Polytechnic University of Timisoara, Romania, by means of XRF analysis with the Niton XL 3 T device. Total solid (TS) and volatile solid (VS) concentration in thesubstrates are determined following the guidelines given by Germany standard protocol VDI 4630. Table 1 shows the results obtained.

The prepared yield Y_2, is calculated as:

$$\boldsymbol{Y = \frac{M_i - M_f}{M_i} \times 100} \quad (3)$$

where M_i represents the mass before calcination and M_f the mass measured after calcination.

Iodine number is defined as the number of milligrams of iodine adsorbed per gram of adsorbate at a residual concentration of 0.02 N. It provides an indication of the micro-porosity of the activated carbon.

The iodine number is determined using the American Society

Table 1
Total solid, volatile solid and total moisture in substrate.

Substrate	TS%	VS%	Total moisture %
Cocoa pod	84.26	63.22	15.70

for Tests and Materials (ASTM) test methods: 100 mg of carbon is introduced into a 100 mL Erlenmeyer flask containing 30 mL of 0.02 N iodine solution; the mixture is stirred for 3 h and filtered; next, 10 mL of the filtrate solution is titrated against 0.005 N sodium thiosulfate solution (Sudaryanto et al., 2006). Equation (4) reports the reaction of iodine with sodium thiosulfate.

$$2NaS_2O_3 + I_2 \rightarrow Na_2S_4O_6 + 2NaI \quad (4)$$

The amount of iodine adsorbed (mg/g) is given by the following relationship:

$$Q = \frac{(C_O - \frac{C_N V_N}{2V_{I_2}})}{M_{AC}} \times M_{I_2} \times V \quad (5)$$

where V_N is the volume of sodium thiosulfate (mL), C_N the concentration of sodium thiosulfate (mol/L), C_O the concentration of the initial iodine solution (0.02 mol/L), V_{I2} the volume of iodine dosed (10 mL), M_{I2} the molar mass of iodine (253.81 g/mol), V the adsorption volume (30 mL), M_{CA} mass of activated carbon (g).

The raw biomass and the produced activated carbon having the best iodine number are characterized by thermogravimetric analysis (TGA), scanning electron microscopy (SEM), X-ray diffraction (XRD) and infrared spectroscopy (FTIR), as better described in the next sections.

2.2.4. Life Cycle Assessment

The environmental impacts of raw material production in the agricultural process as well as its conversion to the final product are investigated by means of the LCA method in order to clearly identify the crucial steps in both phases and improvement needs. The purpose of this LCA analysis is to assess the environmental impacts generated by the use of both agricultural and conversion production steps (e.g., agro-chemicals for cropping and treatment materials and electricity, respectively). The application of LCA to AC preparation process in this work is performed on a laboratory scale, taking into account the consumption of energy, water and chemicals, as well as gaseous emissions involved. LCA is a methodological framework defined by International Organization for Standardization (ISO) standards and International Reference Life Cycle Data System (ILCD) Handbook guidelines (ISO 14044, 2006; ISO, 2006; JRC, 2010). Its aim is to assess the potential environmental impacts and resources use in a cradle to have the perspective, from raw material acquisition, via production and use phases, to waste management. LCA is the standard choice to assess environmental burdens of human dominated systems. All systems' components contribute to environmental impacts by consuming resources, emitting substances into the biosphere and delivering others environmental exchanges. Hence, LCA investigate systems' relations, delivering information about its effects on the environment. LCA results are provided in the form of indicators related to many different environmental impact categories such as, global warming potential, depletion of resources, and toxicological effects among others (Pennington et al., 2004). LCA is a standard tool to investigate the environmental performance of a wide range of human-dominated processes. It is characterized by four stages: 1) definition of goal and scope; 2) inventory analysis; 3) impacts assessment; 4) interpretation.

Different software applications have been developed through the years for the performing of LCA analyses, both free (e.g. OpenLCA) and paid (e.g. GaBi, SimaPro). The LCA presented in this work is performed utilizing the SimaPro software version 9.0.0.49 (https://network.simapro.com/rg), the Ecoinvent database version 3.5 (Wernet et al., 2016), one of the most comprehensive and updated databases available for LCA studies, and the ReCiPe Midpoint (H) v.1.03 method (Huijbregts et al., 2017) for impacts assessment, with a World ReCiPe Midpoint (H), 2010 normalization. SimaPro is a widely recognized, tested and robust, software application for sustainability assessment, allowing users to model complex life cycle analyses in compliance with ISO 14040. It comes with different databases (e.g. Ecoinvent 3.6, Agri-footprint 5, ELCD, USLCI) and methods (e.g. ReCiPe Midpoint and Endpoint, Environmental Footprint, CML) available to use. The chosen Ecoinvent database is currently the world's leading life cycle inventory database, providing well documented process data for thousands of products and adopted by thousands of organization and universities worldwide, thus ensuring a good potential for comparison between different LCA studies. The ReCiPe Midpoint (H) method have been chosen because it allows the investigation of potential of emissions and resource use in different environmental compartments characterized by different representative units, unlike other methods as the ReCiPe Endpoint, applying a long term perspective by accounting for the final damages to the environment, resource use and human health (however introducing a higher uncertainty), or the Environmental Footprint, reducing all the environmental impacts to just the equivalent emissions of CO_2.

Characterized results cannot be compared, due to their different physical units, therefore a normalization procedure is applied. Normalization is a life cycle impact assessment tool used to express characterized impact indicator data in a way that they can be compared among impact categories (Goedkoop et al., 2009; Santagata et al., 2017; Wegener Sleeswijk et al., 2008). The LCA impact categories explored in this study are listed in Table 2.

2.2.4.1. Goal and scope definition. The LCA part of this work is aimed at evaluating the environmental impacts of cocoa in Cameroon, starting with cocoa pods separation after beans fermentation, and the conversion of the separated cocoa pods into activated carbon. The study is conducted at laboratory level, but even in this form it is capable of delivering an idea of the major hotspots throughout the entire chain of processes. The functional unit (FU) chosen, to which all quantities were proportioned is the production of 4 g of activated carbon from an initial amount of 9 g of cocoa pods. This work adopts a cradle to gate perspective, whose boundaries extend from the agricultural phase of cocoa cultivation to their fermentation in wooden boxes to the production of activated carbon at the end of the laboratory phase. The time frame related to data acquisition was 2019. Fig. 2 summarizes the main steps involved.

Table 2
ReCiPe midpoint (H) impact categories.

Impact Category	Label	Unit
Fine particulate matter formation potential	PMFP	kg $PM_{2.5}$ eq
Fossil resource scarcity potential	FSP	kg oil eq
Freshwater ecotoxicity potential	FETP	kg 1,4-DCB
Freshwater eutrophication potential	FEP	kg P eq
Global warming potential	GWP	kg CO_2 eq
Human carcinogenic toxicity potential	HCTP	kg 1,4-DCB
Human non-carcinogenic toxicity potential	HNTP	kg 1,4-DCB
Ionizing radiation potential	IRP	kBq Co-60 eq
Land use potential	LUP	m^2a crop eq
Marine ecotoxicity potential	METP	kg 1,4-DCB
Marine eutrophication potential	MEP	kg N eq
Mineral resource scarcity potential	MSP	kg Cu eq
Ozone formation, Human health potential	OFHP	kg NO_x eq
Ozone formation, Terrestrial ecosystems potential	OFTP	kg NO_x eq
Stratospheric ozone depletion potential	ODP	kg CFC11 eq
Terrestrial acidification potential	TAP	kg SO_2 eq
Terrestrial ecotoxicity potential	TETP	kg 1,4-DCB
Water consumption potential	WCP	m^3

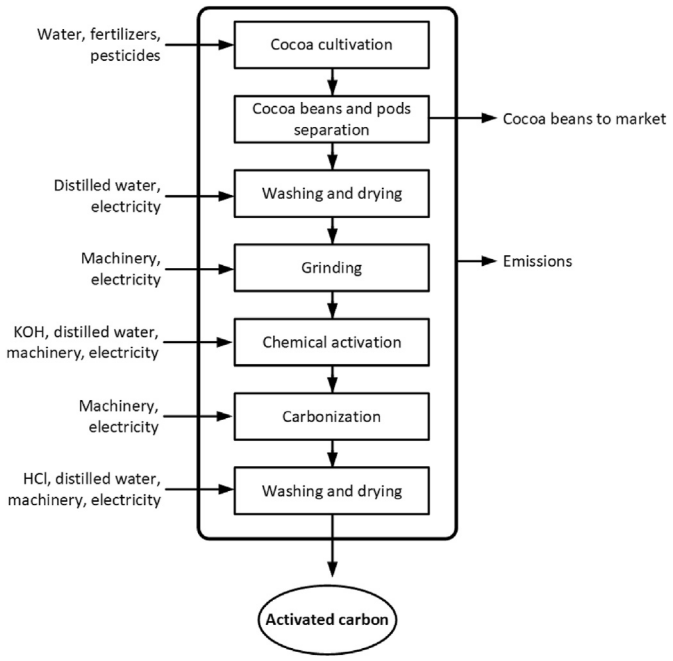

Fig. 2. Flow chart highlighting the main steps of production of activated carbon.

2.2.4.2. Life cycle inventory and impacts. Primary data used within the life cycle inventory (LCI) come from the companies operating the agricultural phase of cocoa cultivation and fermentation (Table 3), and from laboratory data collection for the conversion processes of cocoa pods (Table 4). Impacts and emissions of the agricultural phase are modeled according to Nemecek et al. (2014), developing an agricultural emission estimation method to be used within the framework of the World Food LCA Database, which adopts Ecoinvent as background database (Nemecek et al., 2014). A physical allocation based on mass of achieved cocoa beans and pods after fermentation is applied.

Based on agricultural and laboratory process inventory, the usual Life Cycle Impact Assessment is carried out and final impact critically discussed.

3. Results and discussion

3.1. Optimizing the production of activated carbon

Response Surface Methodology is used as a tool to deduce the optimal values of the concentration of the activating agent, calcination time and calcination temperature, using the yield and the iodine number of the activated carbon as responses.

Table 3
Life cycle inventory of cocoa cultivation and fermentation (1 ha).

Item	Amount	Unit
Input		
Soil occupation	1	ha*a
Water for irrigation	900	m^3
Urea (46% N)	250	kg
(20-10-10) Fertilizer	150	kg
Herbicide	5	kg
Insecticide	10	kg
Output		
Cocoa bean	204	Kg
Cocoa pods	96	Kg
Local emissions		

Table 4
Life cycle inventory of cocoa pods conversion to activated carbon.

	Item	Amount	Unit
Input			
Raw material collection	Cocoa pods	9	g
Grinding/sieving	Electricity	5	kWh
	Water	5	L
Impregnation/chemical activation	Potassium hydroxide	8.415	g
	Electricity	6	kWh
	Distilled water	0.5	L
Washing/drying	Distilled water	6	L
	Hydrogen chloride	500	mL
	Filter paper	10	units
	Electricity	3	kWh
Determination of the iodine number	Potassium iodide	30	g
	Molecular iodine	12.69	g
	Sodium thiosulfate	24.82	g
	Starch	2	g
	Distilled water	1	L
Output			
	Activated carbon	4	g

3.1.1. Mathematical modeling of the responses

Table 5 presents the experimental design matrix using the composite centered method, as well as the response values obtained from the laboratory runs. The observation of the results confirms that the yields by mass vary from 54.97% to 75.68%. As for the iodine number, values between 426.38 and 1035.50 mg/g are observed. All responses are used to develop regression equations of the quadratic models with second order interaction which correlate each response with the three coded variables. This mathematical model takes into account the coded variables, and it is presented as monitoring for the iodine number and of yield during the preparation of the activated carbon. Model equations in coded values for iodine number responses (Y_1) and yield (Y_2) are obtained by replacing each coefficient in equation (2) to obtain equations (6) and (7).

$$Y_1 = 5730.29 - 19.3477X_1 - 690.167X_2 - 94.854X_3 + 0.01908X_1^2 + 0.761778X_1X_2 + 2322.22X_2^2 - 628.183X_2X_3 + 183.635X_3^2 \quad (6)$$

$$Y_2 = 181.717 - 0.441706X_1 + 6.13184X_2 - 8.51974X_3 + 0.000393X_1^2 + 0.091444X_1X_2 - 0.004216X_1X_3 - 75.005X_2^2 + 15.491X_2X_3 + 1.1266X_3^2 \quad (7)$$

The overall adjustment performance of the model is expressed by the correlation coefficient R^2 (Goupy et al., 2006; Tinsson, 2010). When R^2 is close to 1, the experimentally observed values are close to the values predicted by the model. R^2 and adjusted R^2 values related to all the different kinds of activated carbons generated are close to 1. The adjusted R^2 represents the correlation coefficient (R^2) after removing the coefficients not significant for the model. R^2values equal to 0.9272 and 0.9370 respectively for the iodine number and the yield, justify the variability of 92.72% and 93.70% of the response variable. The results confirm the model feasibility to express the iodine number and the yield as a function of the three optimized parameters: temperature, activating agent

Table 5
Experimental design matrix and results for preparation of activated carbons from cocoa pod.

Run	Factors			Y_1 (mg/g) Y_2 (%)					
N°	X_1 (°C)	X_2 (mol/L)	X_3 (h)	Exp. value	Pre. value.	Residuals	Exp. value	Pre. value	Residuals
1	525 .0	0.45	3.0	530.68	626.56	−95.88	**77.72**	77.26	0.46
2	450.0	0.3	3.0	1142.10	1092.18	49.92	75.09	74.77	0.32
3	525.0	0.3	2.0	540.20	543.79	−3.59	74.23	76.07	−1.84
4	600.0	0.6	3.0	1065.96	1013.33	52.63	76.28	77.36	−1.08
5	450.0	0.6	1.0	921.30	863.73	57.57	**55.12**	55.05	0.07
6	525.0	0.6	2.0	534.20	645.37	−11.17	73.30	72.35	0.95
7	525.0	0.45	2.0	**510.14**	433.63	76.51	74.07	71.37	2.7
8	525.0	0.45	1.0	540.32	559.21	−18.89	67.34	68.70	−1.36
9	600.0	0.3	1.0	769.01	762.52	6.49	76.24	76.16	0.08
10	600.0	0.45	2.0	580.30	589.31	−9.01	69.09	68.16	0.93
11	600.0	0.3	3.0	753.79	782.66	−28.87	77.35	77.19	0.16
12	450.0	0.45	2.0	530.40	636.15	−105.75	58.6	60.43	−1.83
13	525.0	0.45	2.0	510.14	433.63	76.51	74.07	71.37	2.7
14	600.0	0.6	1.0	1058.35	1079.58	−21.23	72.24	72.33	−0.09
15	525.0	0.45	2.0	510.14	433.63	76.51	74.07	71.37	2.7
16	450.0	0.3	1.0	867.30	891.24	−23.94	68.22	66.92	1.3
17	450.0	0.6	3.0	**1203.01**	1178.29	24.72	75.56	75.41	0.15

*The negative difference shows that the experimental value is lower than the real value.

concentration and the time of calcination.

3.1.2. Statistical analysis

Results from the analysis of the data using the response surfaces present a second-degree polynomial model. The parameters of the polynomial equation show the relationship between the responses and the factors related to AC production. In this equation, the linear effect of each factor (X_i), their interactions (X_{ij}) and their quadratic effects (X_i^2) on the responses are determined. In the case of this study, significant factors are assessed by means of ANOVA and t -tests. These factors could also be determined by F-tests, which are calculated from the squared sums of the differences between the means. However, the use of the latter involves probability distributions. ANOVA reduces the number of tests required to identify a significant difference in means when comparing more than two groups, thus avoiding unnecessary additional analyses.

The details of ANOVA result are presented in Table 6. The factors are significant if the confidence interval is 95%, which represents a probability less than or equal to 0.05%.

The probability value (P value), equal to less than 0.05, is the most significant among the selected variables. ANOVA results confirm how temperature influences in a linear, quadratic and interactive manner respectively the yield and iodine number of all the kinds of activated carbons obtained. Likewise, iodine number and yield are influenced by the linear and interactive effects of the other factors of preparation.

Furthermore, the F-value can be used to rank these factors in ascending order as: $X_2 > X_3 > X_1$ and $X_2 > X_1 > X_3$ for the yield and iodine number respectively. It is clear how the iodine number is significantly influenced by the concentration of the activating agent.

These results determine the values of constant coefficient α, linear coefficients, interaction coefficients and quadratic coefficients of the different factors. When a coefficient is preceded by a positive sign, it has a synergistic effect on the response, therefore its increase leads to an increase in the response and vice versa. On the other hand, when the sign is negative, the factor has an antagonistic effect: its increase leads to a decrease in the response and vice versa.

An increase in temperature caused a drop in yield. This was expected, as an increase in the decomposition of the biomaterial by the release of volatile matter is activated when the temperature is increased. Other authors had observed similar results of lower yields of activated carbons based on KOH-impregnated precursors (Juang et al., 2001). In addition, heat treatment under an inert atmosphere with prior impregnation of the biomass led to the degradation of the microstructure, resulting in a significant loss of mass. Temperature is also noticed to have a negative influence on the yield, due to an observed significant volatilization of the compounds at high temperature, in accordance with what Gueye et al. (2014) (Gueye et al., 2014) observed with jatropha wood and peanut shells impregnated with phosphoric acid and potassium

Table 6
Analysis of Variance (ANOVA) for iodine number and activated carbon yield.

Source	*Df*	Y_1 (Iodine number)				Y_2 (Yield)			
		SS	MS	F-value	P-value	SS	MS	F-value	P-value
X_1	1	5614.53	5614.53	1.04	**0.3369**	1.217	1.217	0.27	**0.6261**
X_2	1	66438.8	66438.8	12.35	**0.0079***	94.987	94.987	21.00	**0.0059***
X_3	1	155.315	155.315	0.03	**0.8693**	7.536	7.536	1.67	**0.2532**
X_1^2	1	31211.8	31211.8	5.80	**0.0426***	54.466	54.466	12.04	**0.0178***
$X_{1.2}$	1	587.559	587.559	0.11	**0.7496**	8.466	8.466	1.87	**0.2295**
$X_{1.3}$	1	20351.5	20351.5	3.78	**0.0877**	0.800	0.800	0.18	**0.6915**
X_2^2	1	7397.59	7397.59	1.37	**0.2747**	31.692	31.692	7.01	**0.0456***
$X_{2.3}$	1	71030.6	71030.6	13.20	**0.0067***	43.198	43.198	9.55	**0.0271***
X_3^2	1	91375.2	91375.2	16.98	**0.0033***	14.125	14.125	3.12	**0.1374**
Total error	7	79129.2	11304.2			59.7653	8.5379		

$R^2 = 92.72\%$ R^2 adjusted $= 84.52\%$ $R^2 = 93.70\%$ R^2 adjusted $= 85.59\%$.
SS *= Sum of Squares, MS = Mean Square, * significant value, Df = Degrees of freedom.*

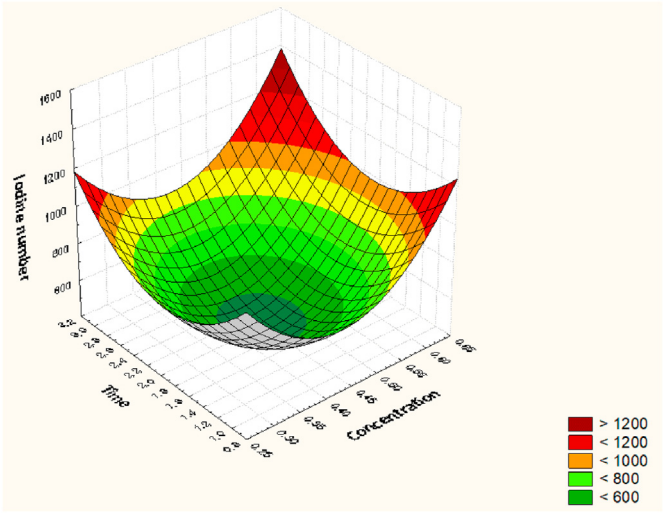

Fig. 3. Response surface for the interaction Time - Concentration as a function of the iodine number.

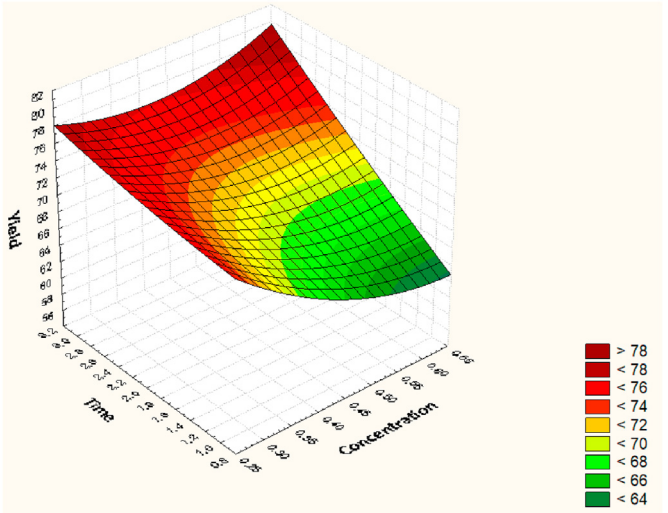

Fig. 4. Response surface for the interaction Time - Concentration as a function of the yield.

hydroxide. Iodine number has its best values at a concentration of the activating agent of 0.6 mol/L, a calcination time of 3 h with a temperature of 450 °C. This is because at low temperature the increase of the activating agent concentration leads to the opening and enlargement of the pores, and consequently a development of the pores which leads to good adsorption of the iodine molecules. Another plausible explanation for this phenomenon is the development of micropores that result from the reaction between the biomass and the high activating agent concentration (Demiral et al., 2016). Results also show that the activated carbons with high concentrations of activating agent have high iodine numbers. This can be attributed to the effect of KOH on biomass during pyrolysis which promotes the volatilization of volatile compounds and inhibits the formation of tar (Juang et al., 2001). The micro-porosity of samples impregnated with KOH is mainly due to the intercalation of potassium atoms in the carbon structure.

3.1.3. Interactions of independent variables

Figs. 3 and 4 present the three-dimensional response surfaces showing the regression equation for the optimization of the three reaction variables, i.e. temperature, concentration and calcination time. Each point in the domain has a response. In this field of study, the value which a response can take can be predicted. The response surface diagrams thus offer the possibility of seeing the evolution of different results and explore the synergistic effect of these factors on the different results.

The results of Table 6 highlights the interaction X_{23} concentration-calcination time as highly significant with a probability $P = 0.0067$ for the iodine number and $P = 0.0271$ for the yield.

The surface diagrams clearly show how the decrease in calcination temperature and the increase in concentration of the activating agent result in an increase in the iodine number. Likewise, increasing the calcination temperature and decreasing the concentration of the activating agent lead to an increase in yield.

3.1.4. Optimal values

It is difficult to optimize the three factors under the same conditions because the fields of interest of the factors are different. Table 7 summarizes the optimal values for yield and iodine number, as well as the related values of the factors, resulting from the superposition of the different curves of the response surfaces. The experimental data are satisfactory and agree with the investigated model. The area of interest is thus identified and it is in this area that the optimal conditions are found.

The performance of activated carbon is evaluated by its ability to adsorb iodine, taken as a reference substance. The iodine number is proportional to the number of micropores. The larger it is, the better the activation level and therefore the better the adsorption capacity. In view of the results obtained, we chose to characterize only activated carbons with large iodine number values.

3.2. Characterization of activated carbon

The raw cocoa pod powder, and activated carbon prepared from it is characterised by a number of techniques:

3.2.1. X-ray fluorescence

The X-ray fluorescence analysis of the cocoa pods aims to determine the contents of heavy metal, presented in Table 8.

The results of the chemical analyses show that cocoa pod biomass is composed of several nutrient elements, i.e. Zr, Pb, Zn, Cu, Fe, Mn, Ca, and K. The one with the highest concentration is potassium with 141.10^3 mg/kg-dry-matter, while the lowest concentration is observed for copper with a value of 66 mg/kg-dry-matter. This biomass does not contain chromium. The high nutrient content of this biomass may be due to its high capacity to absorb and store elemental nutrients in pod tissues (Riggio et al., 2017). The origin of these different elements can be attributed to the nature of the treatment such as irrigation and the fertilizers used during cultivation. These nutrients are also the result of a positive interaction between light and fertilizers depending on the environment in which the cocoa has been grown. It should also be noted that the concentration of these nutrients can be influenced by particle size, humus content, pH, water content, aeration, temperature and root area.

3.2.2. Scanning electron microscopy (SEM)

The SEM analysis was used to determine the morphology of both the activated carbon and the raw cocoa pod. It allowed investigating the size of the pore, shapes and distribution on the surfaces of the adsorbents and precursors. Fig. 5 gives the images obtained.

SEM images shows that AC presents many honeycomb-shaped pores well distributed on the surface. This shows that activation

Table 7
Optimal values of the iodine number and of the yield.

	Iodine number (mg/g)			Yield (%)		
Cocoa pod	X_1 (600 °C) **994.959**	X_2 (0.6 M)	X_3 (1.0 h)	X_1 (600 °C) **74.40**	X_2 (0.56 M)	X_3 (3.0 h)

Table 8
Concentration of macromolecules in cocoa pods expressed in mg/kg dry matter.

Element	Concentration
Zirconium, Zr	5982
Lead, Pb	1066
Zinc, Zn	1396
Copper, Cu	66
Iron, Fe	107
Manganese, Mn	117
Calcium, Ca	6663
Potassium, K	$141 \cdot 10^3$
Chromium, Cr	–

with KOH is effective in creating well-developed pores, thus leading to a large surface area and porous structure. As shown in the cocoa powder image, there was no hole or cavity on the surface of the raw materials. Hence, these cavities only appeared during the activation, resulting from the removal of the impregnating KOH and its derivatives and increasing the surface available for adsorption (Daifullah et al., 2003). The micro porosity observed by the SEM results in the material can also be explained by the fact that cocoa pods contain a much higher content of cellulose and a low content of lignin (Cruz et al., 2012).

3.2.3. IR absorption spectroscopy

The infrared spectra of the raw materials and of the prepared activated carbon are represented in Fig. 6.

An examination of the spectra presents absorption bands of vibrations and deformations that can be attributed to different groupings present in these materials. These bands were identified by the results of the lignocellulosic biomass characterization (Tingaut, 2006; Kenne, 2011).

To appreciate the effect of calcination on the raw material, the spectra of the raw material and activated carbon were compared. The adsorption band of the O–H groups around 3288 cm^{-1} is present on the spectrum of raw material and it is absent from the spectrum of the activated carbon. This disappearance of the O–H vibration band can be explained by the carbonization which is at the origin of the removal of C, H and O atoms in the forms of CO_2 and H_2O (Tchuifon et al., 2014). Indeed, biomass generally contains about 50% carbon, 44% oxygen and 6% hydrogen, its molecule can then be written: $CH_{1.44}O_{0.66}$. Calcination leads to the formation of CO_2 and H_2O molecules according to the equation: $CH_{1.44}O_{0.66} + 1.03\ O_2 \rightarrow CO_2 + 0.72\ H_2O$.

The absorption band at 2925 cm^{-1} assigned to the asymmetric elongation vibration is no longer found in the spectrum of the AC. This indicates that activation removes a significant part of the C–H bond.

3.2.4. X-ray diffraction

The X-ray diffractogram of the AC from cocoa presented in Fig. 7 shows a diffraction peak at $2\theta = 27°$, which is attributed to the presence of graphite carbon (Benamraoui, 2014). This peak corresponds to the highly organized layered structure of graphite, with a distance between layers of 0.34 nm along the orientation (002) (Lua and Yang, 2004). Also, noise signals seen in the spectrum implies that AC has essentially an amorphous structure. Hence, this result can be explained considering that during the carbonization reaction, chemical bonds between the organic compounds in cocoa pods are broken down by the elevated temperatures and the condensation of the active compounds in them. These compounds form layers of typical graphitic planes during carbonization.

3.2.5. Differential calorimetric and coupled thermogravimetric analysis

Results of the thermal analysis of AC resulting from cocoa pods are shown in Fig. 8.

A mass loss of 6.5% is observed between 27 °C and 163 °C, following the exothermic process as shown by the DSC curve, with a negative peak at 81 °C, followed by an endothermic process. A significant loss in mass equals to 83.43% is observed between 248 °C and 538 °C, following an endothermic process with a peak at 440.2 °C. The first observed mass loss may be due to the presence of hygroscopic molecules such as water and other volatile matter or impurities, while the second loss may have been caused by the loss of carbon in the form of carbon dioxide and carbon monoxide (Chubaakum et al., 2015). AC from cocoa pods has excellent thermal stability. The thermo-gravimetric curves highlighting the mentioned losses of mass can be observed, suggest that chemical activation with KOH resulted in a considerable degree of functional formation on the carbon surface.

3.3. Life cycle impact assessment (LCIA)

Table 9 reports the characterized impacts of the agricultural phase for cocoa production (with mass allocation to cocoa pods and cocoa beans), according to the Impact categories listed in Table 2,

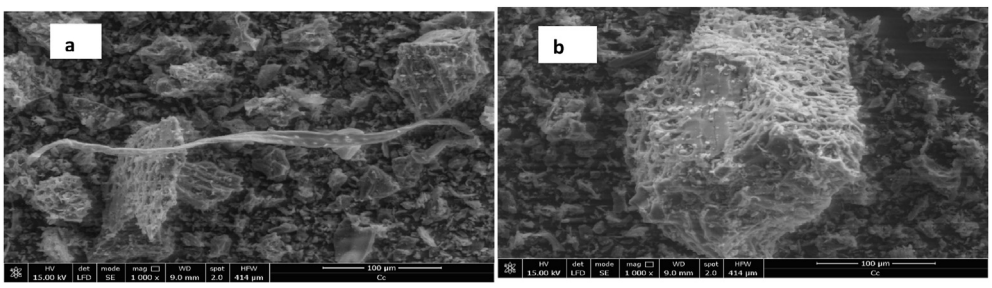

Fig. 5. SEM images of the raw cocoa pod powder (c) and the activated carbon resulting from the cocoa pods (d) (voltage 15 kV and magnification × 1500).

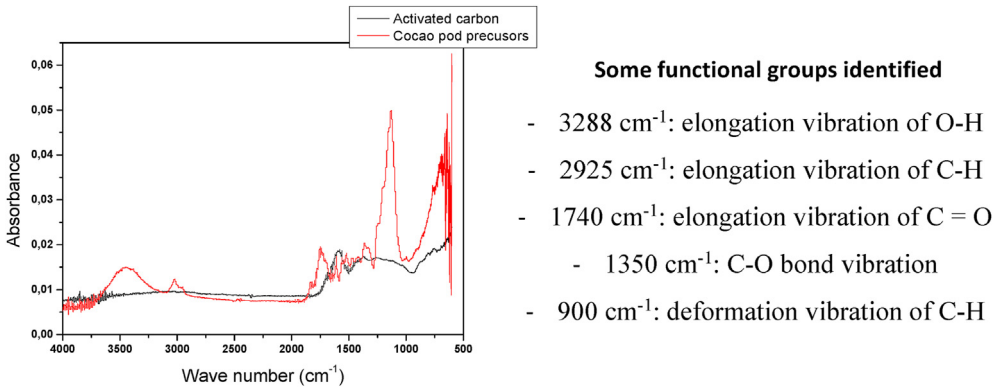

Fig. 6. IR spectrum of cocoa pod powder and activated carbon resulting from cocoa pods.

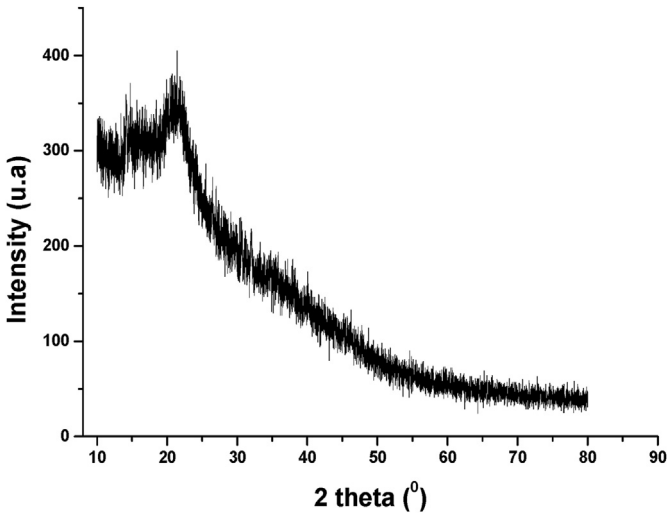

Fig. 7. Diffractogram of activated carbon resulting from cocoa pods.

with reference to the production of 96 kg/ha of cocoa pods.

Table 10 reports instead the characterized ReCiPe midpoint (H) results for the final result at laboratory scale production of 4 g of activated carbon from 9 g of cocoa pods. Of course, values in Table 10 also include the upstream impacts from agricultural phase and must be considered as referring to the entire process as a whole.

Characterized results in Tables 9 and 10 as such do not allow comparison among the different impact categories, which requires normalized values to be calculated (Fig. 9). Some toxicity related categories (METP, FETP, HCTP, HNTP and TETP, with values of 1.4 E+0, 9.4E-1, 9.8E-2, 4.3E-2 and 3.6E-2 respectively) result to be the most impacted within the investigated case study. Toxicity is almost exclusively related to the electrical energy used (average ≈ 93% of contribution). Electricity is also the major contributor to FEP (≈70%) together with distilled water (≈20%). Other impact categories, with smaller, but not negligible normalized impacts, also emerge. Additionally, the functional units of the two evaluations are non comparable, being the agricultural phase referred to 1 ha while the lab phase is sized according to a small

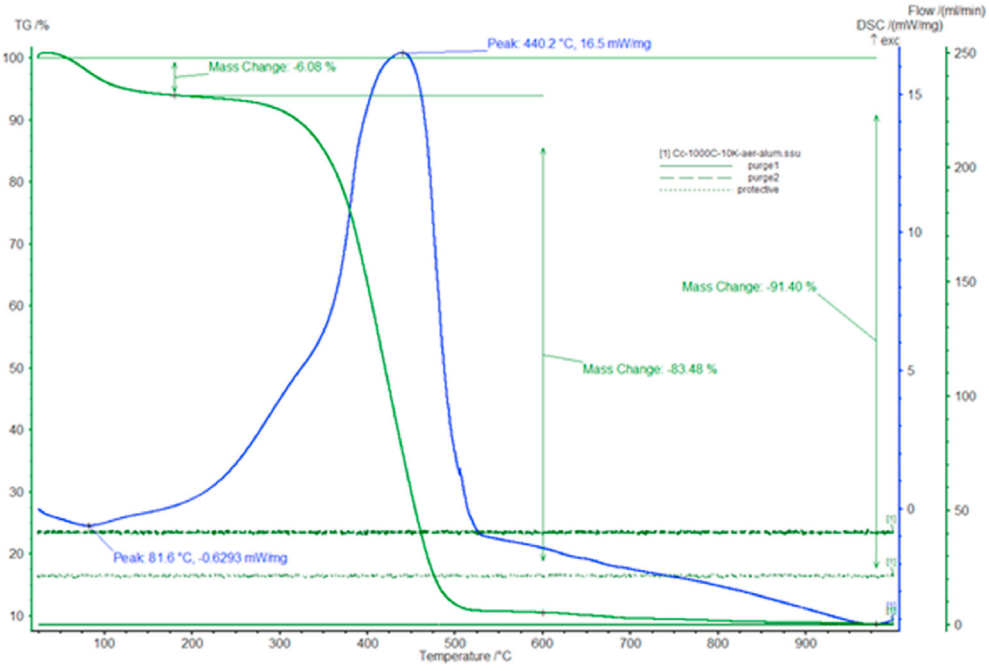

Fig. 8. Thermograms of activated carbon resulting from cocoa pods.

Table 9
Recipe Midpoint (H) characterized impacts for the agricultural production of 96 kg of cocoa pods Impact category.

	Unit	Characterized impacts
PMFP	kg $PM_{2.5}$ eq	3.44
FSP	kg oil eq	114.85
FETP	kg 1,4-DCB	11.97
FEP	kg P eq	0.14
GWP	kg CO_2 eq	768.45
HCTP	kg 1,4-DCB	15.52
HNTP	kg 1,4-DCB	352.38
IRP	kBq Co-60 eq	22.75
LUP	m^2a crop eq	3234.79
METP	kg 1,4-DCB	16.38
MEP	kg N eq	1.08
MSP	kg Cu eq	2.77
OFHP	kg NO_x eq	1.35
OFTP	kg NO_x eq	1.37
ODP	kg CFC11 eq	0.01
TAP	kg SO_2 eq	23.77
TETP	kg 1,4-DCB	1565.12
WCP	m^3	300.59

Table 10
Recipe Midpoint (H) characterized impacts for the production of activated carbon from cocoa pods (reference functional unit: 4 g activated carbon).

Impact category	Unit	Characterized impacts
PMFP	kg $PM_{2.5}$ eq	0.01
FSP	kg oil eq	1.24
FETP	kg 1,4-DCB	1.15
FEP	kg P eq	0.00
GWP	kg CO_2 eq	4.63
HCTP	kg 1,4-DCB	0.27
HNTP	kg 1,4-DCB	6.35
IRP	kBq Co-60 eq	0.10
LUP	m^2a crop eq	0.38
METP	kg 1,4-DCB	1.42
MEP	kg N eq	0.00
MSP	kg Cu eq	0.11
OFHP	kg NO_x eq	0.02
OFTP	kg NO_x eq	0.02
ODP	kg CFC11 eq	0.00
TAP	kg SO_2 eq	0.03
TETP	kg 1,4-DCB	37.22
WCP	m^3	0.06

amount (9 g) of pods.

In order to get a deeper insight into the two production steps, we can identify which are the most important contributing flows to the agricultural step alone and to the entire process by converting values of Tables 9 and 10 into percent values (Figs. 10 and 11), in order to clarify the extent the different inflows contribute to the agricultural phase impacts as well as to the process as a whole. With reference to Fig. 10, the contributions to the agricultural phase impact categories show that an average of 38% contribution over all the impact categories is from all fertilizers combined, an average 33% is from local emissions (mainly derived by the use of fertilizers), an average 26% is from irrigation operations and average 3% from the use of herbicides and pesticides. These results can be compared with some recent LCA application within the agricultural sector, as Saber et al. (2020), reporting that major burdens within rice cultivation are caused by on-farm emissions and diesel use, followed by the use of nitrogen (Saber et al., 2020). Ghasemi-Mobtaker et al. (2020) highlighted that Iranian wheat cultivation presents a significant energy consumption for irrigation and for fertilization, with high contributions to on-site emissions from diesel fuel, mainly used in soil preparation and harvesting (Ghasemi-Mobtaker et al., 2020). Nabavi-Pelesaraei et al. (2018) (Nabavi-Pelesaraei et al., 2018) and Kaab et al. (2019) (Kaab et al., 2019a) show how artificial intelligence is capable of predicting burdens from paddy production, related to the use of diesel fuel and nitrogen fertilizer, and from sugarcane production, presenting high energy consumption within irrigation, diesel fuel and nitrogen used, with high precision. Canola oil production, reported by Khanali et al. (2018) also confirmed the high relevance of the use of nitrogen fertilizer and diesel fuel in the agricultural step regarding global warming, acidification and eutrophication emissions (Khanali et al., 2018). Kaab et al. (2019) showed how the simultaneous use of LCA, Multi-Objective Genetic Algorithm and Data Envelopment Analysis is effective in reducing environmental burdens related to sugarcane cultivation (Kaab et al., 2019b).

Instead, with reference to Fig. 11 (laboratory step also including upstream agricultural production), the major contributor to environmental impacts is the electricity used in the laboratory steps, with an average contribution throughout all the impact categories of almost 70%, with a minimum within LUP ($\approx$9%) and a maximum within FETP ($\approx$99%). A significant contribution also comes from the agricultural phase that produces the cocoa pods, with an overall average contribution of $\approx$12%, with a minimum contribution in METP ($\approx$0.11%) and a maximum in LUP ($\approx$79%). Other significant contributions come from the use of distilled water ($\approx$8% overall), iodine ($\approx$5% overall), potassium iodide ($\approx$1.4% overall) and sodium thiosulfate ($\approx$1.2% overall).

4. Conclusions

This study consisted in obtaining the optimal conditions of the main factors that influence the preparation of AC from cocoa pods in Cameroon and then assessing its sustainability by means of LCA. The effects of various factors influencing the production of AC on the responses were modeled satisfactorily by a quadratic second order expression. Activated carbon prepared at 450 °C for 3 h with an activating agent concentration of 0.6 M shows the greatest adsorption capacity of iodine, which makes it a very promising potential candidate in purification, discoloration and purification of gases.

The purpose of the LCA was to determine the potential environmental impacts associated with the production of cocoa and then transforming the waste pods into activated carbon. The processes involves growing and harvesting the cocoa, separating of the cocoa pods from the cocoa beans after fermentation and then converting the separated cocoa pods into activated carbon. The modeling of the manufacturing process of activated carbon required a large amount of data. These data, whether from the Ecoinvent database, literature, estimates and laboratory measurements helped to determine the environmental profiles of the process. Important parameters such as the consumption of electricity and raw materials have been identified. The use of electricity and other chemicals was found to be environmentally harmful in the context of manufacturing activated carbon. It emerges from this analysis that the major contributor to the environmental impact is the electricity used in the preparation stages, where the average contribution in all impact categories was almost 70%.

However, LCA results should be very carefully interpreted, as this laboratory conversion process might differ from a fully implemented industrial one, where the most suitable machinery and the right amounts of energy and materials would be more efficiently used. In this work, LCA contributions from the agricultural phase are rather minor. The most probable reason for this is the small quantity of cocoa pods used in what essentially is a laboratory chemical analysis. The average cocoa cultivation and cocoa seeds and pods separation in Cameroon broadly relies on human labor, unlike other regions (i.e. Ghana), where this kind of products

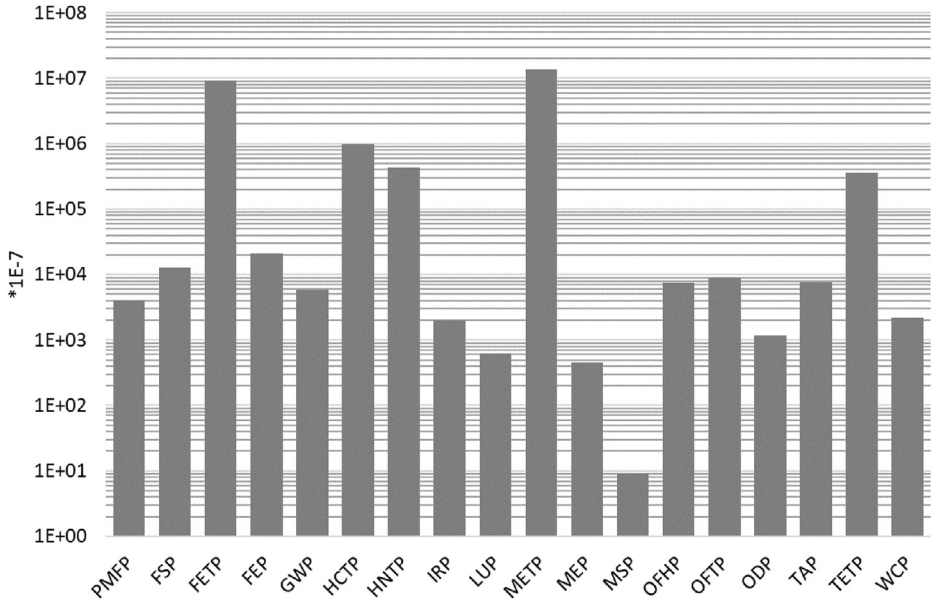

Fig. 9. World ReCiPe Midpoint (H), 2010 normalized impacts for the production of 4 g of activated carbon (*1E-7).

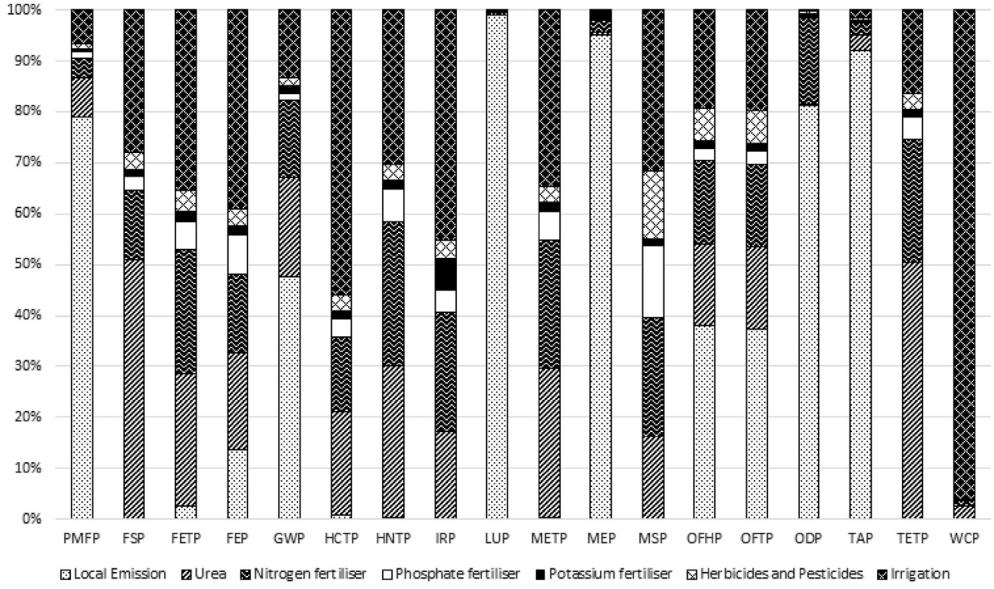

Fig. 10. Percentage contribution of input flows to the ReCiPe Midpoint (H) characterized impacts of the agricultural production of 96 kg of cocoa pods.

are cultivated in an intensive way. The development of a likewise way of cultivation of cocoa coupled with a more efficient conversion process of the conversion step would be reflected in more appropriate, and likely lower, LCA impact indicators. From an environmental point of view, the added value of such conversion process resides in the recovery and use of a biomass that is commonly disposed of by means of incineration, releasing into the environment harmful greenhouse gases and particulates. A thorough investigation of benefits and constraints must be implemented to assess and compare the common disposal methods of this kind of biomass and its conversion, taking into account also the disposal of exhausted activated carbon.

The results of this work suggest that mastering the main influencing factors in the production of activated carbon and its manufacturing process is not only essential for obtaining a microporous carbon, but also economically and environmentally profitable. Thus, cocoa pods can be considered as a feasible material for the preparation of activated carbon by chemical activation from an economic and environmental point of view. The environmental benefit of the production of AC from cocoa pods lies mainly in the protection of the biosphere by re-evaluating a material destined to landfilling or incineration by using it to produce high value AC. The species of cocoa pods used, the pretreatment and activation techniques and the lack of certain data during the cultivation of its fruits present some limits in terms of quantifying the environmental impacts, and future work will be essential to improve data reliability.

Another important outcome from this paper is represented by the before mentioned limitations due to the laboratory scale of the investigated process. This is even more important regarding LCA

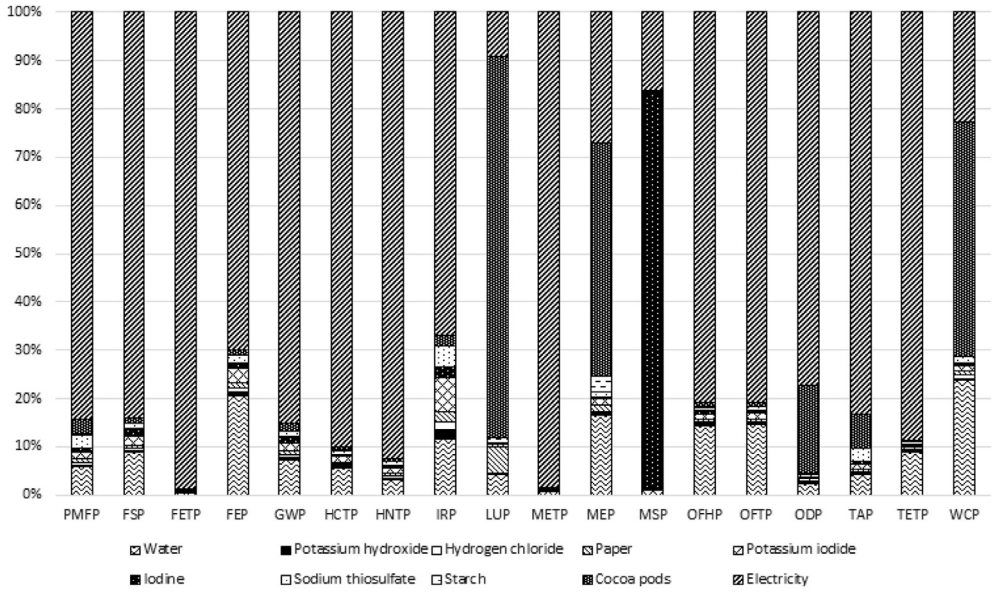

Fig. 11. Percentage contribution of input flows to the ReCiPe Midpoint (H) characterized impacts of the laboratory production of 4 g of activated carbon.

results, that are expected to be widely different in terms of percentages of the various inputs to the final impacts. In a real life application, it may be expected a major contribution coming from the agricultural step, almost non-significant in the presented lab scale analysis. Policy and management recommendations could only be done based on the real life industrial application of the presented conversion process. However, the lab to industrial upscaling would not be possible without a chemical and environmental assessment as the one performed in this work.

CRediT authorship contribution statement

Rufis Fregue Tagne Tiegam: All authors listed have provided a significant contribution to the paper, and all those who are qualified to be authors are listed in the author byline. **Donald Raoul Tchuifon Tchuifon:** All authors listed have provided a significant contribution to the paper, and all those who are qualified to be authors are listed in the author byline. **Remo Santagata:** All authors listed have provided a significant contribution to the paper, and all those who are qualified to be authors are listed in the author byline. **Paul Alain Kouteu Nanssou:** All authors listed have provided a significant contribution to the paper, and all those who are qualified to be authors are listed in the author byline. **Solomon Gabche Anagho:** All authors listed have provided a significant contribution to the paper, and all those who are qualified to be authors are listed in the author byline. However, due to their equal contribution to the development of the paper, we kindly ask to include both Solomon Gabche Anagho and Sergio Ulgiati as corresponding authors. **Ioana Ionel:** All authors listed have provided a significant contribution to the paper, and all those who are qualified to be authors are listed in the author byline. **Sergio Ulgiati:** All authors listed have provided a significant contribution to the paper, and all those who are qualified to be authors are listed in the author byline. However, due to their equal contribution to the development of the paper, we kindly ask to include both Solomon Gabche Anagho and Sergio Ulgiati as corresponding authors.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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