OBTENTION OF ACTIVATED CARBON BY REUTILISATION OF OIL PALM RESIDUES: A CIRCULAR ECONOMY CASE STUDY

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ABSTRACT

Activated carbon is a highly porous material with favourable properties for adsorption, as a water and gas purifier, and in removing molecules. In the African oil palm industry, a considerable amount of vegetable waste, including palm kernels, is generated at the end of the production chain, which degrades surrounding environmental conditions. Therefore, reusing this waste is essential to contributing to circular economy approaches. This work aims to evaluate the quality parameters of activated carbon produced from palm kernels following Ecuadorian standards through chemical treatments in the laboratory for the valorisation and use of this resource. For this purpose, this work suggests three phases that include: (i) a review of relevant literature; (ii) chemical treatment through activation with H_3PO_4 (20% and 40%); and (iii) quality tests following Ecuadorian standards. The results showed that moisture content, total ash and density were within permissible ranges according to Ecuadorian quality standards. In addition, adsorption tests using methylene blue showed a final concentration of approximately 2.05 ppm and a maximum of approximately 15 ppm, considering an initial value of 100 ppm, with similar effectiveness to commercial versions. Finally, an economic analysis showed a production cost of 3.038 USD/kg and an estimated market price of 4.50 USD/kg, making it highly competitive in the Ecuadorian market. In conclusion, this work constitutes a proposal for the final use of agro-industrial waste to contribute to the circular economy of the African oil palm, obtaining a highly competitive product according to its capacity for adsorption of domestic and industrial pollutants, as well as its monetary value with possibilities for strategic marketing that projects on a small and medium scale.
Keywords: agroindustry, chemical activation, biomass, adsorption, carbonisation, oil palm.

1 INTRODUCTION

Circular economy is a leading concept for managing resources at the end of any production chain, aiming to promote the revalorisation of waste into new products [1], [2]. Additionally, it is recommended that resource use pursue the search for economic profitability of revalorisation to guarantee the activity as sustainability over time and to be an incentive for its permanence [3]–[5].

Many industries produce biomass at the end of their production chain [6], [7]. One example is the oil palm industry, where a plant residue known as ‘shell’ is generated after oil extraction [8]. Like many plant residues, this resource produces biodiesel [9] and lightweight aggregate in cement concrete [10]. However, another example of the reuse of hulls is in producing activated carbon [11].

This product is an amorphous solid with a considerable internal area and porous volume, ideal for reagent adsorption uses [12]. In an early stage, activated carbon comes from biomass

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to which carbonisation processes (e.g. carbonisation and hydrothermal carbonisation) are applied to develop the porous structure, culminating in the extraction of the biochar [13], [14]. A second step involves the development of the porous structure by physical and chemical processes [13].

Similarly, activated carbon as a product also comes from other plant materials, including rice husk, sawdust, banana peel, and even materials such as cardboard obtained from thermocouple sensor tubes [15]–[17]. Related literature suggests that the type of biomass used for production significantly influences the properties of the final product [18]. For example, waste plum stones, pine sawdust, and horsetail herb-activated carbons have shown exceptional adsorptive characteristics for heavy metal ions such as Pb (II) and Cu (II) [19].

Although there are other materials with recognised purification capacities (e.g. zeolites, kaolin and activated muds) [17]–[20], activated carbon, in addition to having attractive characteristics such as a large internal surface area, regeneration capacity, with its possibility of reutilisation, several times [21], and versatility in the final objective of its production [22], as the potential to chemical modifications to eliminate specific contaminants (e.g. dyes and odours) [23], in addition, although the scrubbing capacity of activated carbon may be less effective compared to other products on the market (e.g. zeolites and activated muds) [24], from an economic point of view, it makes it an option when the intended use of the material requires keeping strict control of the final cost of its application [25].

Therefore, this work presents a case study for reusing African palm crop residues as a development opportunity for economic and environmental benefits at the end of the production chain. For this reason, this research poses the following question: Is the activated carbon obtained from African palm hulls a resource for purifying pollutants and with prospects for commercialisation? Therefore, the objective is to evaluate the quality parameters of the activated carbon obtained from African palm shells using chemical treatments in the laboratory for its valorisation and contribution to the circular economy.

2 MATERIALS AND METHODS

This proposal focused on using African palm residues to produce activated carbon in three phases, including: (a) raw material adequation; (b) chemical treatment; and (c) economic analysis and quality testing to evaluate the product's effectiveness (Fig. 1).

2.1 Stage I: Sample adequation

The first stage involved preparing the sample for processing and determining its capacity to obtain activated carbon. The sample used corresponded to 500 g of shell from an oil palm collected from the waste of an oil palm industry. The pre-treatment of the sample began with removing impurities accompanying the material in its original state (e.g. dust, residual oil, fibres). After cleaning, two parts carried out the drying: the first part was the exposition with sunlight for 72 hours, while the second part performed dry in an oven at a temperature of 110°C in the Hydrocarbon Laboratory of the Faculty of Natural Sciences and Mathematics of ESPOL University, Ecuador.

2.2 Stage II: Chemical treatment

The second stage comprises chemical treatment, divided into two parts. First, it involved chemical activation, impregnating H_3PO_4 at 20% and 40% concentrations in the 500 g sample. The second part consisted of carbonising the sample at 500°C. Finally, the result of

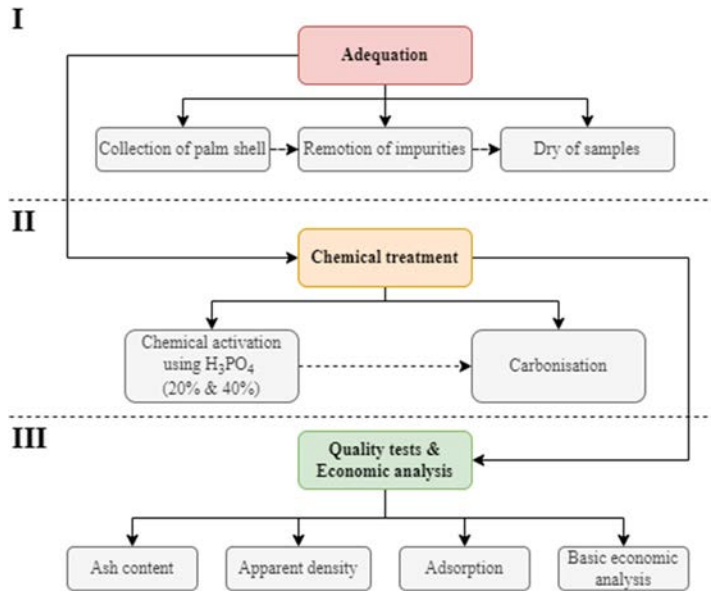

Figure 1: Methodological flowchart for the obtention of activated carbon by oil palm shell.

the previous processes underwent a second washing to remove residual acid and then crushed to a granular size (6–10 mesh).

2.3 Stage III: Quality tests and economic analysis

Finally, the last stage comprised different tests to measure the quality properties, according to the Ecuadorian standard INEN 1983:2013. Table 1 shows the tests on two commercial samples compared to two samples of African palm shell.

Similarly, a basic economic analysis determined the viability of commercialisation as a final product. In addition, this analysis also made projections such as net present value and internal rate of return to obtain the payback period of a proposed initial investment. Furthermore, the work considered estimated values for machinery prices, annual operating costs, data from recent years on the palm oil industry, and activated carbon prices in the Ecuadorian market.

3 RESULTS

Table 2 summarises the initial conditions of the shell mass used to obtain activated carbon, obtaining a quantity of 3,310 g.

3.1 Chemical activation

Chemical activation by impregnating phosphoric acid determined an acid and organic matter impregnation rate of 2:1, so 500 ml of acid solution was required for a 250 g sample. Specifically, the dilution of H_3PO_4 and the mass of the acid solution obtained allowed us to establish an experimental impregnation rate of 2.22 and 2.54, with the solutions of the mentioned reagent at 20% and 40%, respectively.

Table 1: List of quality tests done for every activated coal sample.

Test name	Description	Equations
Ash content	A crucible is calcined in a muffle at 650°C for 1 hour, cooled and weighed. Simultaneously, 10 g of dry sample is weighed and dried at 150°C. Then, a 1 g dried coal sample is incinerated at 650°C for 4 hours, cooled, and weighed. The process repeated until the mass is constant, and then we calculated the ash content.	$\%C = \frac{m_1 - m_c}{m} x100$ where: %C = total ash content m = sample mass (g) m_1 = crucible mass with ashes (g) m_c = empty crucible mass (g)
Humidity content	The sample is dried in an oven at 130°C. The water lost by heating is known by comparing the initial and final masses.	$\%H = \frac{P_i - (P_f - P_c)}{P_i} x100$ where: P_i = initial mass of activated carbon P_f = final crucible and carbon mass P_c = dry crucible mass
Apparent density	This test determines the quality of the carbon in terms of its adsorption capacity using the pore volume ratio.	$\rho_{\text{apparent}} = \frac{m \times (100 - \%H)}{V \times 100}$ where: m = sample mass in grams $\%H$ = humidity content V = test tube volume in cm^3
Decolourant capacity	This test determines the adsorption capacity of activated charcoal and the concentration of the final solution. It uses a 100 ppm methylene blue solution to design a calibration line using six solutions with different concentrations. After adsorption testing in an activated carbon column for 16 hours, a UV spectrophotometer calculated the absorption measurement.	$\text{Decolorizing power} = \frac{15}{10} x \frac{V_2}{M}$ where: V_2 = volume of methylene blue consumed M = pulverised activated carbon mass

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Table 1: Continued.

Test name	Description	Equations
Langmuir isotherm	A test that measures the adsorption capacity is based on the volumetric analysis of a sample of acetic acid 0.5 M in six Erlenmeyer flasks, each with 2 g of activated carbon. After 15 minutes of stirring and rest, it was titrated with NaOH 0.1 N using phenolphthalein as an indicator.	$q = \frac{q_o c}{K + c} \rightarrow \frac{1}{q} = \frac{K}{q_o}\frac{1}{c} + \frac{1}{q_o}$ where: $\frac{1}{q}$ = inverse of the amount of solute adsorbed per unit mass of adsorbent $\frac{1}{c}$ = inverse of the solute concentration in the solution K = adsorption equilibrium constant q_o = maximum adsorption capacity
BET surface	Reveal the maximum relationship between moles of adsorbed acid and grams of activated carbon using the molecular area of acetic acid ($\sigma = 2.1 \times 10^{-19}\mathrm{m}^2$), per absorbed molecule.	$A = \sigma \times N_A \times \left(\frac{x}{m}\right)_{\mathrm{Max}}$ where: A = superficial area in m^2/g N_A = Avogadro number ($\frac{6.022x10^{23}\mathrm{molecule}}{\mathrm{mol}}$) $\left(\frac{x}{m}\right)_{\mathrm{Max}}$ = maximum constant
Iodine number	It is the mass in milligrams of iodine adsorbed per gram of activated carbon when the concentration of the remaining filtrate iodine is 0.02 N. This test helps quantify porosity, which shows the initial sample's activation degree.	$\text{iodine number} = \frac{V}{m} \cdot f$ $\frac{V}{m} = \frac{A - (2.2 \times B \times V_t)}{m}$ $C = \frac{N_2 \times V_t}{50}$ $A = N_1 \times 12{,}693$ $B = N_2 \times 126.93$ where: v/m= milligrams of iodine adsorbed per gram C = normality of the residual filtrate f = correction factor V_t= volume of 0.1 N thiosulfate consumed N_2 = normality of the standard 0.1N sodium thiosulfate solution N_1 = normality of the standard 0.1N iodine solution

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Table 2: Pretreatment of shell oil palm.

Parameter	Initial shell mass	Dry shell mass	Pretreated shell mass
Weight	5,000	3,500	3,310
Humidity	30	–	–
Useful fibres (%)	–	5.42	–

However, the process performance showed a mass loss route of 250.13 g to 78.36 g, while chemical activation at 20% did from 205.02 g to 102.89 g. Thus, the quantified yield in percentage for each acid concentration used represented 31.33% for activation at 40% and 41.15% for activation at 20%.

3.2 Ash content

On the other hand, according to the INEN 1987 standard, the total ash content should not exceed 12%, with a 7.89% and 4.15% content for the activated samples at 40% and 20%, respectively. The process of washing and crushing may have influenced this percentage of ash, as the presence of irregular granular forms encourages the generation of ash with the traces of sprayed coal because due to the nature of obtaining the shell as raw material for the activated coal process, there may be soil particles that by being composed of moisture caused by volatile compounds and solids dissolved, can influence the results in this parameter.

3.3 Humidity content

The moisture content obtained was 7.56% and 10.23% for the 40% and 20% phosphoric acid samples, respectively. Simultaneously, Commercial samples 1 and 2 showed 13.28% and 11.11%. Both own samples and one of the commercial samples comply with the INEN 1985 standard, which recommends a maximum of 12% for the case of granular coal. The favourable values are explained by the fact that the coal obtained was insoluble in water, which facilitated the removal of water contained in the surface when heating a fireplace at 130°C. Additionally, wash water's evaporation was crucial to remove traces of hydroxide and acids.

3.4 Apparent density

The results within this parameter were 0.5104 g/cm^3 and 0.5167 g/cm^3 for activated carbon at 40% and 20%, respectively. Compared with the two commercial samples, Commercial 2 reached values like the previous ones (0.515 g/cm^3), while Commercial 1 reached a value of 0.5833 g/cm^3. These values are within the range permitted by the INEN 1986 standard by exceeding the value of 0.2 g/cm^3 for granular coal and showing that the three samples with the lower apparent density exhibit the highest porosity (see Table 3).

3.5 Adsorption analysis

Fig. 2 shows the adsorption analysis using the reagent (methylene blue). It also highlights that both cases (own and commercial samples) exhibited similar behaviour in the adsorption of contaminants, with a differentiation in the initial volumes. Additionally, Fig. 2 shows that as the volume of the 'pollutant' increases, its concentration is more significant, indicating the

Table 3: Apparent density results for all samples of activated carbon.

Sample	Test tube mass (g)	Test tube + carbon mass (g)	Carbon mass (g)	Humidity (%)	Volume (ml)	Apparent density (g/ml)
H_3PO_4 40%	32.7955	38.3100	5.5195	7.5600	10	0.5104
H_3PO_4 20%	32.7955	38.5600	5.7648	10.2300		0.5167
Commercial 1	32.7955	39.5300	6.7371	13.2800		0.5833
Commercial 2	32.7955	38.5900	5.7945	11.1100		0.5150

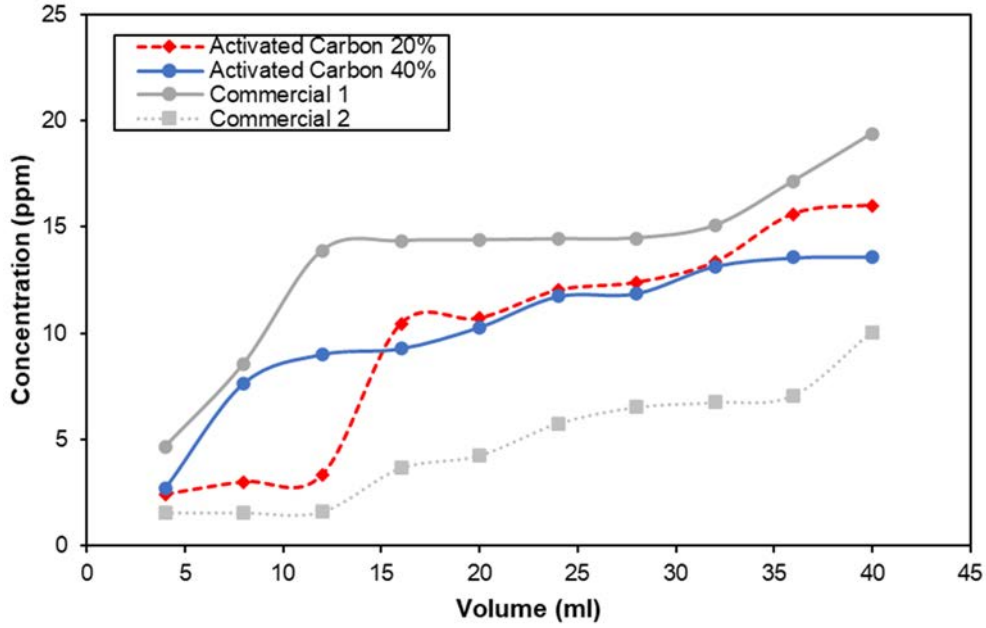

Figure 2: Absorbance plot of methylene blue concentration in activated carbon at 20% and 40% of H_3PO_4 and Commercial 1 and 2.

saturation of the coal and its respective adsorption capacity. In such a way, H_3PO_4 40% palm shell activated carbon and Commercial 2 obtained the best results by being the ones that most absorbed the test reagent.

3.6 Langmuir isotherm and BET surface

Fig. 3 shows the adsorption isotherms obtained from volumetric analyses of the concentration of acetic acid in equilibrium and the adsorbent–adsorbent ratio for the four samples analysed.

This graph served for the calculation of the BET area (Table 4), derived from the previous parameter (Langmuir constant), which highlights the difference between values obtained for each sample, where the standard recommends a minimum value of 500 (m^2/g), which is only met by both commercial coals, suggesting slight effectiveness in pollutants with considerable particle size.

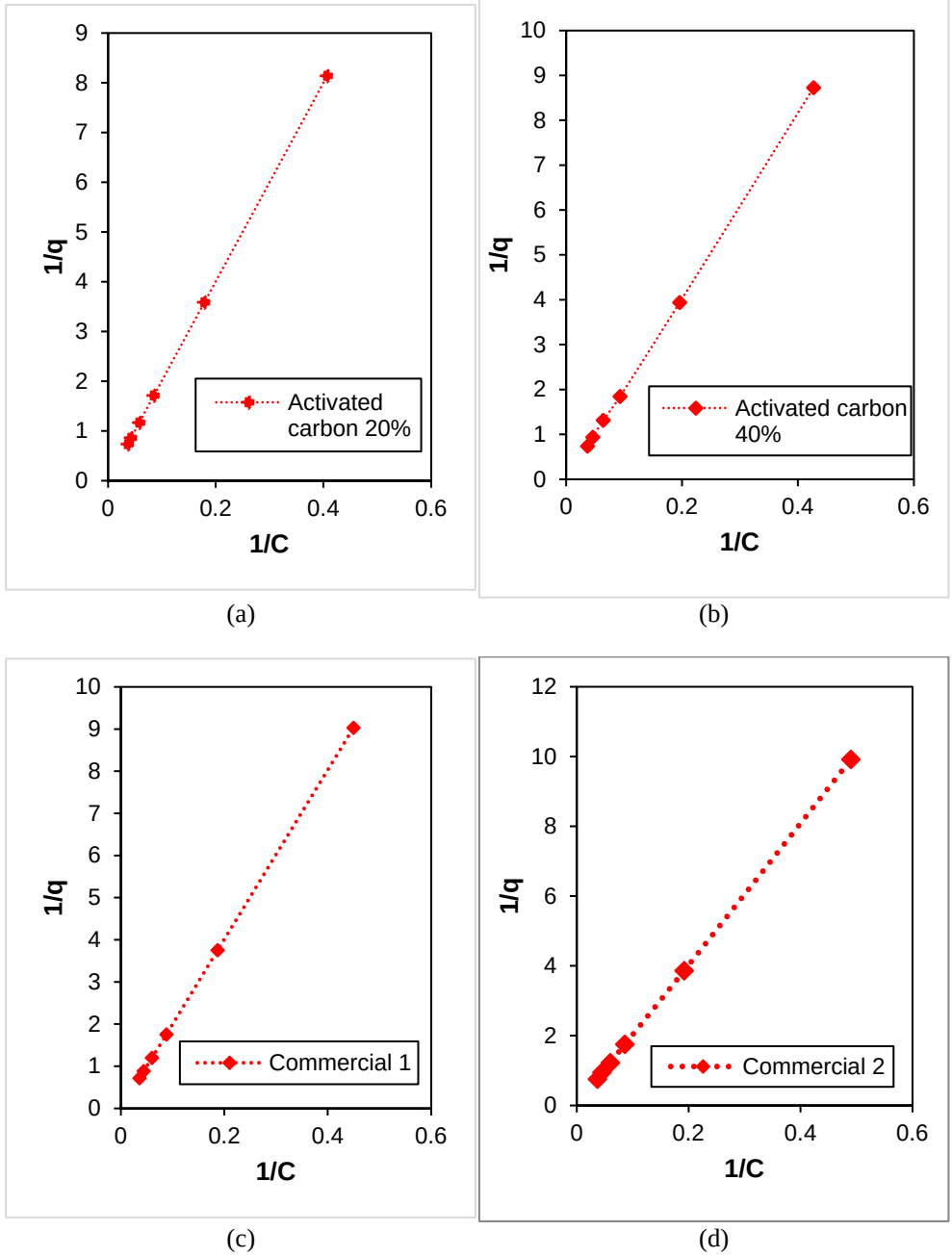

Figure 3: Langmuir isotherm for the samples of: (a) activated carbon at 20%; (b) activated carbon at 40%; (c) Commercial 1; and (d) Commercial 2. Abbreviations: 1/q = inverse of the amount of solute adsorbed per unit mass of adsorbent; 1/C = inverse of the solute concentration in the solution.

Table 4: BET area for all the samples analysed in this work.

Sample	Langmuir constant $\left(\frac{x}{m}\right)_{Max}$	$\sigma(m^2)$	Surface area $\left(\frac{m^2}{g}\right)$	BET surface $\left(\frac{m^2}{g}\right)$
H_3PO_4 40%	0.0033	2.1×10^{-19}	417.32	339.22
H_3PO_4 20%	0.0032	2.1×10^{-19}	404.68	432.84
Commercial 1	0.0045	2.1×10^{-19}	569.08	568.29
Commercial 2	0.0043	2.1×10^{-19}	543.79	531.18

3.7 Iodine number

This test recorded the consumed volume of 0.1 N sodium result to calculate the iodine adsorption capacity per mass of activated carbon and the surface area associated with this number. The results of Table 5, like the previous parameter, suggest that only commercial coals can be adequate according to the standard analysed.

Table 5: Iodine number results for all samples of activated carbon.

Sample	Sample weight (g)	Volume consumed (ml)	Adsorbed iodine (mg/g)	Area (m^2/g)
H_3PO_4 40%	2.00	23.50	346.00	344.09
H_3PO_4 20%	1.20	28.70	440.13	437.71
Commercial 1	1.60	14.00	576.31	573.14
Commercial 2	1.60	18.00	539.00	536.03

Finally, the pollutant removal capacity under the various analysed methods showed that activated coal with the largest surface area was activated carbon at 40% of H_3PO_4 with 417.12 m^2/g (Langmuir), 344.09 m^2/g (iodine) and 339.22 m^2/g (BET). In contrast, 20% H_3PO_4 activated carbon showed values of 404.68 m^2/g (Langmuir), 437.71 (iodine) and 432.84 (BET). It is worth noting that the values of the three tests in the two types of activated carbon are close to the surface area required by the INEN regulation (500 m^2/g). Thus, the resulting values suggest limited effectiveness in clearing large particle-sized contaminants; however, they help remove dyes, given the properties exposed in the methylene blue tests.

3.8 Basic economic analysis

Table 6 summarises an economic analysis that allowed a brief analysis of the financial viability of its marketing in Ecuador. The summary references an industrially activated coal plant for nutshell and cost assessment approaches and a price per tonne of palm shell of 72 USD.

Because laboratory experimentation allowed us to obtain a shell conversion to activated coal between 30% and 40%, the study assumed a plant conversion of 30%. Thus, out of 10,000 kg/day, it would be possible to obtain 3,000 kg of activated coal daily, equivalent to 960 metric tons per year. Considering these considerations, this work received an estimated fixed capital investment of $4,652,070.74 for equipment and capital costs, with an annual operating cost of $2,916,460.00. So, the value of activated coal will be USD 3.038, highly competitive compared to similar obtained from other types of raw material (e.g. coconut

Table 6: Brief economic summary of activated carbon from palm oil plant shell commercialisation in Ecuador.

General data – Demand for activated carbon	
Oil palm exports – Ecuador (2019)	187,494,000 kg
Total biomass generated	749,976,000 kg
Shell generated	56,248,200 kg
Percentage of used shell	5.68
Activated carbon consumption	2,263,492 kg/year
Economic projections	
Equipment costs	$3,259,070.74
Capital costs	$1,393,000.00
Total fixed capital investment	$4,652,070.74
Total annual operating cost	$2,916,460.00
Estimated annual production of activated carbon	960,000.00 kg
Estimated cost of palm shell activated carbon	$3.038/kg
Suggested price	$4.50/kg
Price range in Ecuador	$4–7/kg
Net current value (NCV)	$1,232,231.53
Internal rate of return (IRR)	28%
Return on investment (ROI)	3.02%
Payback period (PBP)	1.59 years

shell). For example, at the national level, product values fluctuate between USD 3.8–7/kg, and similarly, at the international level, where value has prices ranging from USD 1.75 to USD 3.15/kg, which means that the marketing has a net return of USD 140,492.54 per year.

Considering the shell price, it suggests a trading value of $4.50 to guarantee a return on investment of 3.02%, equal to a net annual return of USD 140,492.54 per year. The above data helped to denote profitability within the project by obtaining a net current value (NCV) of $1,232,231.53 and an internal rate of return (IRR) value of 28%. Finally, these data allow an estimated return period of 1.59 years, corresponding to the time the proposal recovers its fixed capital investment.

4 CONCLUSIONS

This work obtained activated carbon by chemical activation, with a process yield of 41.15% and 31.33%. In addition, a precursor-activator impregnation rate of 2.22 and 2.54 was retained for the starting material at 20% and 40% H_3PO_4, respectively.

The analysis of the decolourising power showed that both samples (20% and 40%) can purify pollutants. In contrast, the 40% sample stood out as having a similar capacity to one of the commercial samples (Commercial 2). On the other hand, the other adsorption tests carried out under different tests (Langmuir, iodine, BET) showed values within the permissible ranges according to the local standard (INEN) by the commercial samples. However, the samples obtained from palm hulls did not meet the minimum required by the standard. Therefore, this study suggests that carbon is better functional for purifying pollutants with small molecules (e.g. dyes).

From an economic point of view, the profitability of the production of activated carbon from palm shells can be attractive in the Ecuadorian market because the suggested price

($4.50/kg) is lower compared to the range of values of the material in the market. Thus, considering an approximate investment of USD 4 million, the projections can achieve an internal rate of return of 28% in a payback period of 2 years. It is worth mentioning that this economic analysis is essential, but further study is needed to determine values that are close to reality.

Finally, testing with other activated carbon methodologies (e.g. microwave radiation) is recommended, aiming to increase the final product's porosity, which is decisive in capturing pollutants. Similarly, other tests, such as phenol, ketoxime, tannin, and butane adsorption, are suggested to find optimal applications in addition to those identified by the study.

ACKNOWLEDGEMENTS

The authors would like to thank Faculty of Natural Sciences and Mathematics (FCNM, acronym in Spanish), Centro de Investigación y Proyectos Aplicados a las Ciencias de la Tierra (CIPAT, acronym in Spanish), and Faculty of Engineering in Earth Sciences (FICT, acronym in Spanish), from ESPOL University, Ecuador, for their support in the development of the present scientific work.

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