Sustainable Motor Oil Recycling Process using Banana Peel (*Musa Paradisiaca*) for Fe ion Adsorption

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Abstract— *Due to the inadequate disposal of materials containing potentially toxic elements, the concern with the preservation of the environment has increased. Biosolvent materials have been used as an alternative material to treat waste. The objective of this paper was to test the efficiency of banana peel (musa paradisiaca) for the adsorption of Fe ions present in spent motor oils. The banana peels were dried, crushed and sieved (16 mesh). The oil used was collected from the disposal tanks in gas stations in the city of Uberaba, Minas Gerais State, Brazil. The analyses were carried out in batch mode, under agitation, to guarantee contact between the adsorbent and the oil. After contact, oil and adsorbent were separated by centrifugation or by vacuum filtered. The process of liquid-solid separation by centrifugation influenced the results due to the action of the centripetal force causing the separation of the ions by drag but resulted in a removal efficiency of 33.43 to 94.53% of Fe iron removal from the oil, depending on the treatment time. Using vacuum filtration, the process had an efficiency of 38.58%.*

I. INTRODUCTION

According to the annual report by the National Association of the Automachine Vehicles Manufacturers (Associação Nacional dos Fabricantes de Veículos Automotores - ANFAVEA), 91 million vehicles were produced worldwide in 2019 (ANFAVEA, 2020) and it is estimated that there are 1.4 billion vehicles in use. Lubricating oil is an essential for the operation of these motor vehicles, as it plays a role in reducing friction, heat transfer and corrosion resistance (Li et al., 2020) and approximately 40 million tonnes of lubricating oil are required annually to service the world fleet (Lam et al., 2016). Lubricating oils are complex and contain various additives, such as antioxidants, defoamers, anti-wear, thickeners, corrosion protection, detergents, dispersants,

pour point depressants and extreme pressure additives (Yash et al., 2015).

According to the Brazilian technical standard, NBR 10.004 (ABNT, 2004b), lubricating oil is classified after use as a dangerous waste, due to its characteristics of flammability, corrosivity and reactivity. After use, lubricating oils contain degraded additives and undesired substances that can cause adverse effects that also justify the classification as dangerous waste (Lam et al., 2016).

It is a fact that a large part of these lubricants in the post-use phase are discarded and cause pollution of the environment, contaminating soils and groundwater reaching food, harming public health. In Ordinance No. 685, of August 27, 1998, ANVISA establishes maximum levels of contaminants (mycotoxins, inorganic

contaminants, pesticide residues, veterinary drugs and migrants from packaging and equipment in contact with food) in foods that pose risks to human health (ANVISA., 1998).

With the proposal of contributing to the reduction of the inappropriate disposal of industrial contaminants, this work was developed with the purpose of treating automotive oils in a sustainable manner. Treatment of used oils is a subject of few studies; however, it covers a wide area of research.

As a concept of sustainability, the practices of 6R (recover, remanufacture and redesign, recycle, reduce and reuse) promote actions that reduce the impacts generated by the incorrect disposal of this waste (Jawahir and Bradley, 2016). In the context of the remanufacturing of lubricating oil, the 6R concepts contribute to the mitigation of the environmental impact caused by this dangerous waste, which can contain soot, polycyclic aromatic hydrocarbons and metal ions present in oil additives (Peng et al., 2018).

In addition to mitigating the impacts caused by the disposal of this waste, it is worth mentioning that there are economic advantages in the practices of the 6R concept, as explained by Jawahir and Bradley (2016). These purification practices can be conducted by many techniques, such as adsorption (Miyagi et al., 2003),

extraction (Mohammed et al., 2013), hydrotreating (Ramasamy et al., 2007), coagulation, oxidation and vacuum distillation (Shri et al., 2014).

In the context presented, this study aims to test the efficiency of the *Musa Paradisiacal* banana peel in adsorbing Fe ions present in used automotive oils, by employing an adsorption process. For this purpose, this article will be structured as follows: In section 2, a literature review on the concept of Adsorption that will serve as a methodological basis for the study proposed in this article. Section 3 presents the methodology for the present study and the results are presented in section 4. Contributions and conclusions are discussed in section 5.

II.   LITERATURE REVIEW

In addition to the basic oil degradation products, the original additives are also present in the used oil and have not yet been consumed (Li et al., 2020), as well as metals from engine and machine wear, such as lead, chromium, barium and cadmium (Peng et al., 2018). Various contaminants, such as water, unburned fuel, dust, and other impurities are also encountered (Pinheiro et al., 2017). Table 1 indicates the main contaminating elements found in lubricating oil and the concentration ranges encountered.

Table 1. Main elements and contaminants in lubricating oils (Concawe, 1996)

Elements	Origen	Limits (ppm)
Ba	Detergent additives	<100
Ca	Detergent additives	1000 - 3000
Mg	Detergent additives	100 - 500
Zn	Anti-wear additives	500 - 1000
Fe	Engine degradation	100 - 500
Cr e Ni	Engine degradation	Traces
Al, Cu e Sn	Bearing	Traces
Si	Additives/water	50 - 100
S	Base oil / combustion products	0.2 – 1%
Light hydrocarbons	Fuel	5 – 10%
Poly aromatic hydrocarbons (PAH)	Incomplete combustion	<1000

The adsorption process involves fixation of soluble liquids and/or gases on thesurface of a solid (Fiyadh et al., 2019). The adsorption operation exploits the ability of given

solids to concentrate certain substances present in liquid or gaseous solutions at their surface, allowing them to be

separated from the other components present in these solutions (McCabe, 1986).

These substances are called adsorbates (Yang et al., 2015).There are two main types of adsorption: physisorption and chemisorption (Nascimento et al., 2014). Physisorption occurs when the adsorbent and adsorbate are joined by Van Der Waals forces and chemisorption occurs when the adsorbate molecules are bonded to the surface of the adsorbent by chemical bonds (Fiyadh et al., 2019). McCabe (1986) complements that these processes can also be defined as physical adsorption or chemical adsorption and explains that physical adsorption occurs only by adsorption of the component (solute) of interest in the pores of the adsorbent, whereas in chemical adsorption there is a reaction between the adsorbent and the adsorbate.

There are several methods for removing metal ions, including ion exchange, solvent extraction, reverse osmosis, precipitation, coprecipitation and adsorption. Activated carbon and other carbon-based adsorbents are widely used because they are efficient in removing (via adsorption) metal ions. Due to the high cost of synthesis and regeneration of activated carbon, there is a continuous search for low-cost adsorbents, which demonstrate efficiency in the adsorption process (Ferreira, et al., 2015).

In the relevant literature, several different types of adsorbent have been used for the removal of heavy metals. These are separated as adsorbents of non-organic or organic origin. For the first group, modified chitosans, manganese oxides and extracellular polymeric substances (Yang et al., 2015), activated sodium titanate nanofibers (Sounthararajah et al., 2015b), among others, can be exemplified. For the organic group, peanut shells (Brown et al., 2000), peat and sewage sludge ash (Ho and McKay, 1999), granular biomass (Hawari and Mulligan, 2006), fly ash (Weng and Huang, 2004), landfill clay (Ghorbel-Abid and Trabelsi Ayadi, 2015), activated carbon (Sounthararajah et al., 2015a), and sugarcane bagasse (Ferreira, 2015), can be mentioned.

It is a fact that all these adsorbents suffer from some limitations, such as low adsorption capacity and low levels of effectiveness and efficiency in removing heavy metals, and have their efficiency measured by adsorbed components that are concentrated on the outer surface of the solid. Therefore, the larger the surface area available, the more favorable it will be for the adsorption process. The particles that act as the adsorbent, present between 5 to 30% of adsorbed area, reaching 50% in exceptional cases (McCabe, 1986).

Yang et al. (2015) explains that the quality of this process depends on the adsorption capacity, which in turn depends on the characteristics of the adsorbent surface and how it interacts with specific pollutants. For example, surface load, surface area and functional groups can produce different levels of activity for different pollutants.

When considering adsorption processes and adsorbents of organic origin, three important studies can be cited:

a) Brown et al., (2000) evaluated the potential of peanut shell pellets to capture metal ions from wastewater and compared their performance to that of raw peanut shells and a commercial quality ion exchange resin. The 90% removal of Cu^{2+}, Cd^{2+}, Zn^{2+} and Pb^{2+} was verified.

b) Ferreira (2015) described the efficiency of Cu^{2+} and Cr^{3+} adsorption from liquid effluents, using the sugarcane bagasse ash. The results indicated an average removal efficiency of 97.3% for Cr^{3+} and 96.4% for Cu^{2+}, and this material can be applied as an adsorbent in industrial effluents.

c) Abdel-Jabbar et al., (2010) investigates the recovery of automotive lubricating oil used by different adsorbent materials, such as shale egg powder, date palm almond powder, and acid activated palm kernel powder by adsorption processes and obtained results in the extraction of asphaltenic contaminants and metallic oil residues.

III.   METHODOLOGY

3.1. Infrared Spectroscopy (FTIR)

As presented by Concawe (1996) in used automotive oils it is possible to verify a concentration of 100 to 500 ppm of Fe ions. With the objective of verifying whether the samples collected for this study contained Fe ions or other elements resulting from their decomposition in the used automotive oil, the methodology of Infrared Spectroscopy (FTIR) was used to identify traces of Fe or derived by-products of Fe.

3.2. Banana peel treatment

As a starting point to carry out the tests to determine Fe in the samples, according to the methodology of Skoog et al. (2013) and Nascimento et al. (2014), the banana peels, raw material for the adsorbent in this study, were placed in an oven at a temperature of 60º C for a period of 48 hours and, subsequently, crushed in the knife mill. The crushed material was passed through a granulometric sieve, selecting the particles of interest with 16 mesh.

3.3. Calibration curve for concentration calculations

Then, as initial tests, using the dilutions made from the standard $Fe(NO_3)_3$ (1000 mg/L), it was possible to plot the calibration curve (Figure 1), which made it possible to obtain the iron concentrations in the samples under study.
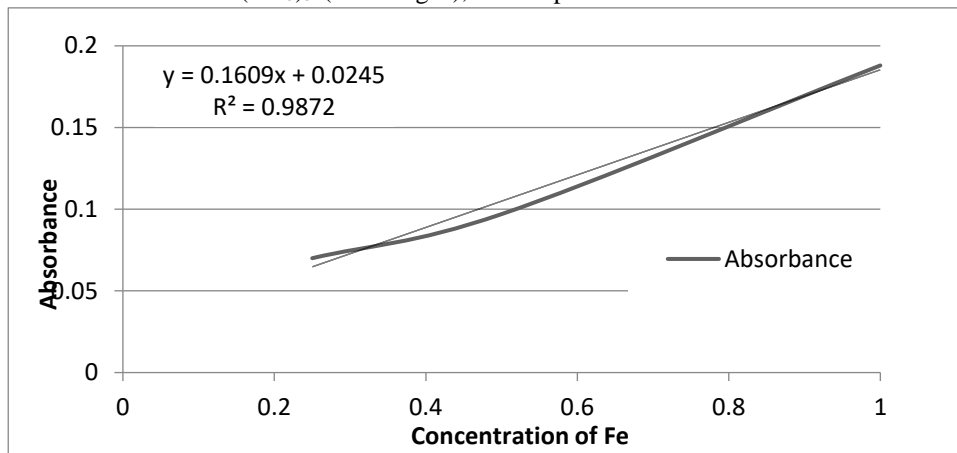

Fig.1: Calibration curve for concentration calculations

According to the methodology of Skoog et al. (2013) for Fe determination in samples, the calibration curve was generated from the standard iron (III) solution (1000mg / L) diluting to the concentrations (mL/L) of 0.80, 1.60, 2.40, 3.00, 20.00 and 4.00, following reading spectrometer. All samples were treated at room 25℃.

3.4. Motor oil treatment

The used oils studied here were collected from gas stations in the city of Uberaba-MG. After collection of all material, the oil samples were homogenized.

Before starting the adsorption tests using the peel and used oil, the base level of Fe ions in unused oil was determined by adding 7.2×10^{-3} g of iron (III) nitrate ($Fe(NO_3)_3$) per gram of unused automotive oil. The dilution of $Fe(NO_3)_3$ is very slow, and the mixture was stirred for 24 hours to ensure the homogeneity of the mixture. These initial tests were important to determine the ideal contact times between the adsorbent and the oil for the adsorption process to occur.

3.5. Variation parameters

Treatment of the used oil was performed by varying parameters such as adsorbent mass, oil mass and the contact time, with the objective of increasing the efficiency of adsorption of the iron, present in the oil, using the peel.

The banana peel and the oil were homogenized by magnetic stirring for determined contact times, defined as homogenization time (T_h). Subsequently, the separation step was performed by vacuum filtration (filter paper diameter of 15µm). For the purpose of comparison, the separation was also performed using a centrifuge for 5 mins at 2000 rpm.

After treatment, the banana peel (2 g) and oil (2 g) from each sample were calcined in a muffle furnace and, subsequently, the quantification of iron present was performed.

IV. RESULTS AND DISCUSSION

4.1. FTIR analysis of oil samples

With the results obtained using FTIR, it was possible to obtain the spectrum given in Figure 2. The bands corresponding (1) to 487 cm^{-1} and (2) to 644 cm^{-1} can be associated with literature values (Namduri and Nasrazadani, 2008), proving the presence of Fe by-products, originating from engine wear.
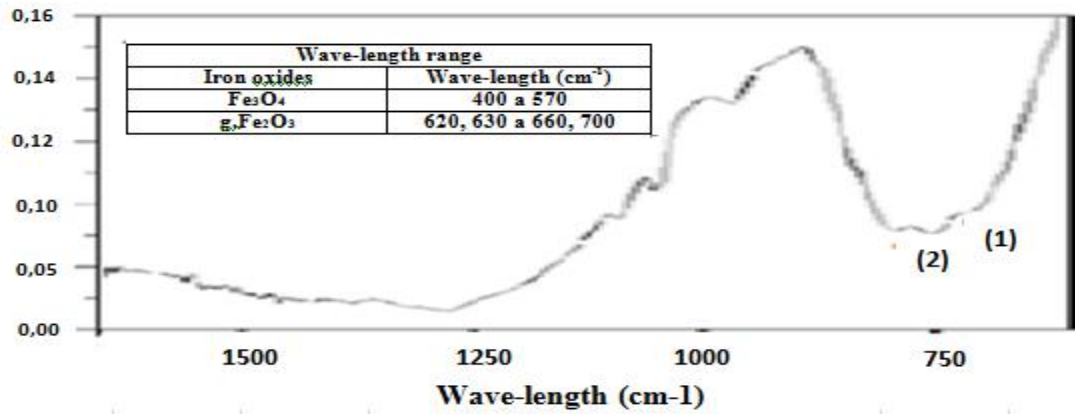
Fig.2: FTIR analysis of oil samples

4.2. Fe absorption efficiency by A_d

To analyze the effect of homogenization time (T_h) on the absorption efficiency of Fe by the adsorbent (A_d), centrifugation tests were performed on oil samples (40 g), with a centrifugation time of 20 min, in which the T_h was varied by 0.12, 1.0, 2.0, 4.5 and 24.0 hours and the weight of A_d at 2.0, 5.0 and 10.0 g, and in this analysis the centrifugation time was fixed at 20 min. In these tested conditions, the concentrations of Fe in the oil samples, the Absorbances and the% of Fe in the oil samples were obtained and are shown in Figure 3. Each analysis was performed in triplicate, and the results are given in Table 2.
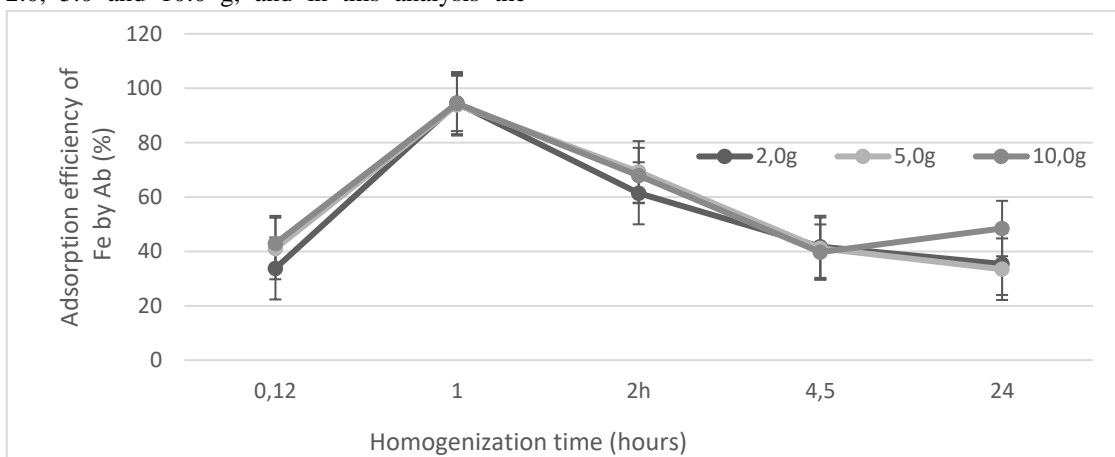

Fig.3: Variation of Fe adsorption efficiency as a function of T_h of A_d mass.

The values obtained in the tests were based on concepts of analysis of variance and hypothesis testing for an analysis of equality of means for a significance level of 5%. In this case, when the statistical significance (p) is greater than 0.05, the variables are considered statistically equal; when it is less, they are considered statistically distinct. For these calculations, Analysis of Variance (ANOVA), and Student's t test were employed. The significance level of the averages was 0.0089, being characterized as statistically distinct, with 95% reliability.

Table 2. Residual iron concentration in samples varying Th and Ad mass

Treatment data		[Fe]	% Fe
A_d (g)	T_h (h)	(mg Fe/Kg of oil)*	
0.0	-	75.11	100.00
2.0	0.12	49.78	66.28
2.0	1.00	4.10	5.47

2.0	2.00	29.02	38.64
2.0	4.50	43.80	58.32
2.0	24.00	48.53	64.62
5.0	0.12	44.27	58.95
5.0	1.00	4.57	6.09
5.0	2.00	23.09	30.78
5.0	4.50	44.22	58.88
5.0	24.00	49.99	66.56
10.0	0.12	42.91	57.14
10.0	1.00	4.19	5.59
10.0	2.00	24.11	32.11
10.0	4.50	45.27	60.28
10.0	24.00	38.75	51.60

As the objective of analyzing the interference of Th in Fe absorption efficiency by Ad in percentage and this is shown in Figure 3. Note that Th was efficient for values close to 1h, and the values for 2.0, 4.5 and 24 h the values obtained were unsatisfactory. This fact is explained by McCABE (1986) in which the desorption process, which is the passage of the solute particles to the liquid phase, that is, the inverse adsorption process can occur. For the value of 0.12 hours the result was also unsatisfactory, and this fact can be explained by the low time given for the adsorption process to occur.

4.3. Influence of Ad on Fe adsorption

It can also be noted in Figure 4 that when the Ad mass was varied, the results obtained are very similar, showing a significant difference for the treatment with 2.0 g of peel. The fact that there is little significant difference in the residual amount of iron, suggests a more detailed analysis of the contribution of Ad mass to the absorption efficiency of Fe by A_d.
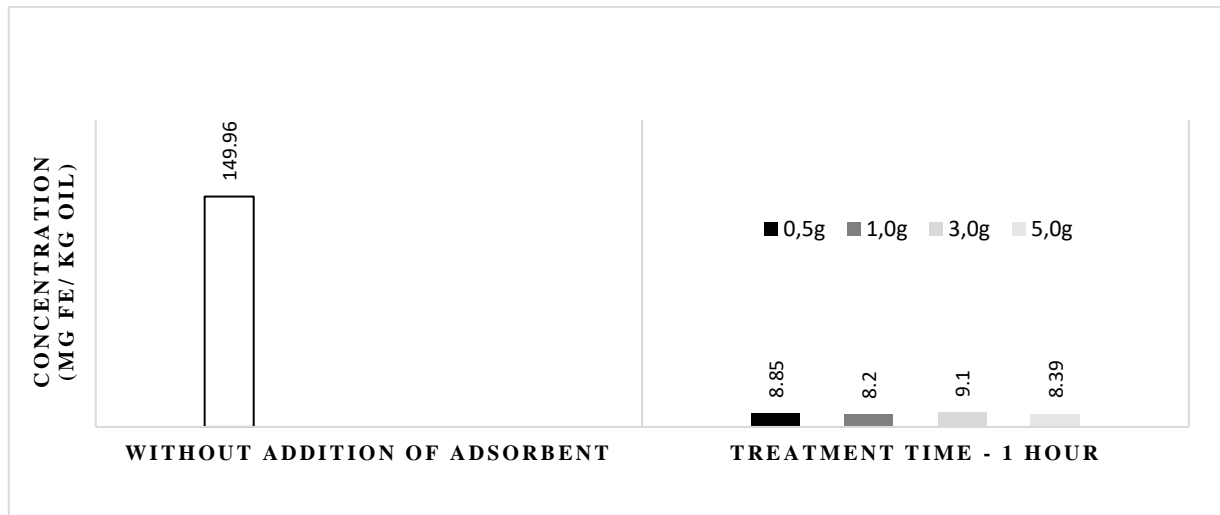

Fig.4: Residual iron concentration after centrifugation, varying the A_d mass.

To prove that the amounts of Ad would not influence the adsorption process, another test was carried out on samples of 40 g oil, varying the amount of peel by 0.5, 1.0, 3.0 and 5.0 g and setting T_h to 1 h and the centrifugation time at 20 min. These results are presented in Table 3 and in Figure 5, confirming that, as in observed in Figure 4, there is significant effect of A_d mass on the adsorption process.

Table 3. Residual Fe concentration varying the A_d mass and fixing Th at 1 h.

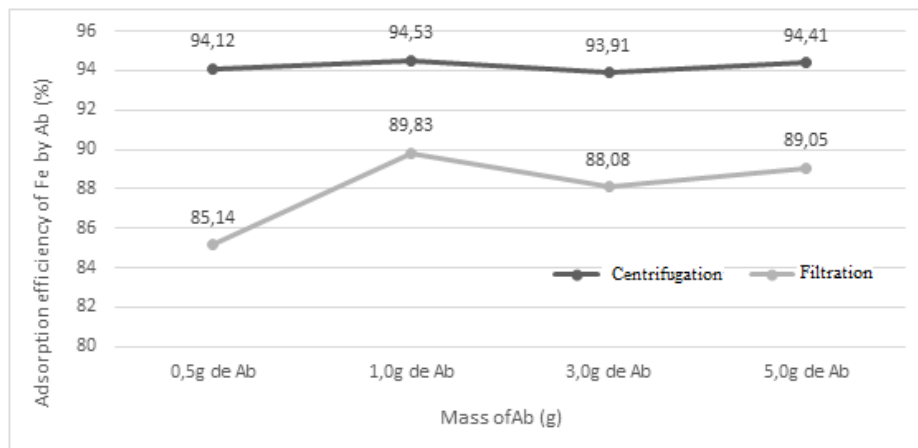| A_d (g)	[Fe] (mg Fe/Kg of oil)	Absorbance	% Fe
-	149.96	1.231	100
0.5	8.82	0.096	5.88
1.0	8.20	0.090	5.47
3.0	9.136	0.098	6.092
5.0	8.390	0.092	5.595

Fig.5. Comparison of absorption efficiency between centrifugation and filtration.

4.4. Residual iron concentration in relation to the centrifugation time

Subsequently, now with the objective of analyzing the results in which the Th values close close to 0.12 h and with variations in A_d mass, tests were performed in which samples of oil (40 g), varying the amount peel in 0.5; 1.0; 3.0 and 5.0 g and setting the Th at 0.12 h and the centrifugation time at 20 min. These results are shown in Table 4. Note that the results were similar, which indicates that low T_h does not contribute to the absorption process, even at low Ad mass values, such as 0.5 or 1.0 g. Facts explained by the low contact time between the adsorbent and the adsorbent.

Table 4. Residual iron concentration varying the Ad mass and fixing Th at 0.12 h.

A_d (g)	[Fe] (mg Fe/Kg of oil)	Absorbance	% Fe
-	30.88	0.273	100
0.5	18.21	0.171	58.95
1.0	17.94	0.168	58.01
3.0	18.00	0168	58.29
5.0	17.65	0.166	57.14

After analyzing the data, where it was noted that the ideal times for the adsorption process are values close to T_h of 1 hour, as shown in Table 3. For the subsequent analyses, this value will be fixed, and the A_d masses varied at 0.5, 1.0, 3.0 and 5.0 g. To optimize time and verify the influence of the centrifugation time on the results, the centrifugation time was set at 2 min and 20 min. The results are shown in Table 5. The difference in the

percentage of iron adsorption between treatments was very small, but with a noticeable emphasis on the longer centrifugation time. This fact points to the need for further

analysis to verify the contribution of the centrifugation time to the adsorption process.

Table 5. Residual iron concentration varying centrifugation time

A_d (g)	Centrifuge time (min)	[Fe] (mg Fe/Kg of oil)	Absorbance	% Fe
-	-	30.88	0.273	100
1.0	2	6.84	0.030	4.82
1.0	20	8.20	0.090	5.47
5.0	2	6.20	0.025	4.39
5.0	20	8.39	0.092	5.59

From Table 5, it cannot be said that centrifugation time was the main parameter to influence the results. However, with these results, some questions arose about the methodology used for separating the Ad and about the solubility of iron nitrate III in oil.

4.5. Comparison absorption efficiency between centrifugation and filtration

According to Green and Perry (2019), the centrifugation process occurs due to the centripetal force that arises through the rotation of the material around a central point. The content of the flask exerts an equal and opposite force on the walls of the container. As a result, all material is subject to two forces: gravity (downward) and centrifugal (horizontal).

Considering the theory of Green and Perry (2019), it was suspected that when the samples were

centrifuged, the entire peel (along with the Fe ions) was dragged to the bottom of the Falcon tube. Thus, as the oil collected for calcination was at the top, consequently, it would be exempt or with a small concentration of iron.

Bearing in mind that the solubility of iron nitrate II in oil is low, even maintaining the agitation for 24 hours, no analysis was carried out to ascertain this homogenization (Figure 5). However, tests were carried out directly with burnt oil, and for comparison criteria treatments were performed with centrifugation and vacuum filtration.

In a new research strategy for this study, it was decided to compare the results obtained by centrifugation and vacuum filtration. Thus, oil (40 g), Th of 1 h with separation by vacuum filtration or by centrifugation for 20 min was performed, varying A_d (Table 6).

Table 6. Fe concentrations, by filtration and centrifugation, varying the Ad mass.

A_d (g)	Separation	[Fe] (mg Fe/Kg of oil)	Absorbance	% Fe
--	--	149.96	1.231	100
0.5	Centrifugation	8.82	0.096	5.88
1.0	Centrifugation	8.20	0.090	5.47
3.0	Centrifugation	9.136	0.098	6.09
5.0	Centrifugation	8.390	0.092	5.59
0.5	Filtration	22.28	0.244	14.86
1.0	Filtration	15.25	0.167	10.17
3.0	Filtration	17.87	0.195	11.92
5.0	Filtration	16.42	0.180	10.95

In Figure 5 can be seen that the centrifugation process proved to be able to remove more Fe ions compared to vacuum the filtration process. Note the differences in efficiencies between filtration and centrifugation are constant for 1.0, 3.0 and 5.0 g, however for the 0.5 g mass there is an increase in this difference, possibly caused by the small amount of Ad.

4.6. Contribution of the centrifugation

In a new analysis strategy for this study, now with the objective of determining the contribution of the centrifugation time to the adsorption process, the amount of iron present in the oil and in the peel after centrifugation was determined. For this analysis the mass of Ad was fixed at 10 g, the Th was 1 h and centrifugation time was 2 min.

The results are given in Table 7. Three types of sample were studied, the first being 40 g of oil without added adsorbent (Ad = 0). For the 2nd sample, oil (40 g) with 10 g of adsorbent was vacuum filtered and after this procedure, a solid mass (solid material from filtration) and a liquid mass (liquid material from filtration) were obtained, and from these two by-products the values of Fe concentrations were measured. For the 3rd sample of oil (40 g), 10 of adsorbent was added and centrifuged for 2 min and a solid mass (solid centrifugation material) and a liquid mass (liquid centrifugation material) was obtained, and the Fe concentration determined.

4.7. Comparison of residual iron concentrations obtained by centrifugation and filtration

Analyzing the treatment carried out by vacuum filtration and centrifugation, it was possible to observe that the iron concentration in the oil decreased and the peel increased. This result appears to be coherent, and it is possible to conclude that a small interval of time is sufficient for peel-oil separation.

Analyzing the oils, it is noted that the iron concentration when it is separated by centrifugation (0.06 mgFe / kg oil) is much lower than the oil concentration by filtration (6.89 mgFe / kg oil), being able to conclude that the centrifuge process works as described by Green and Perry (2019).

The theory of Green and Perry (2019) applies to the results obtained with the adsorbents, because in the treatment by centrifugation, the iron ions were dragged to the bottom of the Falcon tube by the centripetal force or by the drag caused by the movement of the peel. Ad from filtration showed a higher percentage of iron than those resulting from the centrifugation process.

Table 7. Residual iron concentration in oil and peel samples treated for 5 min with separation performed by vacuum filtration and centrifugation for 2 min.

Tratamento	[Fe] (mg Fe/Kg of oil)	Absorbance	% Fe
Ad = 0	14.170	0.138	100.000
Liquid material from filtration	0.684	0.030	4.823
Liquid material from filtration	13.275	0.131	93.686
Liquid material from filtration	0.062	0.025	0.439
Liquid material from filtration	8.788	0.095	62.019

This result can show that the shells analyzed after the centrifugation process were influenced by the centrifugal and gravitational forces. On the other hand, the results obtained from the shells separated by filtration showed the concentration of iron that was adsorbed, showing the efficiency of the process and reaching the objective of the work (Figure 6).
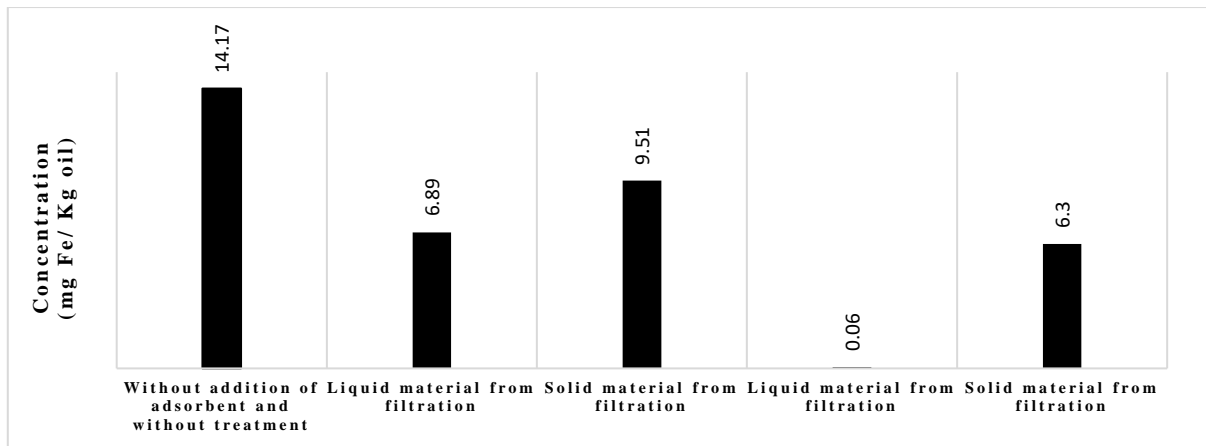
Fig.6: Residual iron concentration in samples separated by vacuum filtration and centrifugation.

4.8. Efficient parameters for Fe adsorption

Finally, to determine up to which moment the adsorption process is efficient, new treatments were performed using Ad equal to 0.5 and 10 g, in oil samples (40 g), treated by vacuum filtration. Th values were 5, 30 and 60 min. These results are shown in Table 8, and it is possible to observe the adsorption kinetics.

Table 8. Residual concentration in mg Fe / Kg oil varying Th and Ad, separated by vacuum filtration.

Tratamento com filtração	Concentração	Absorbância	% Fe
Sem tratamento	44,189	0,380	100,000
Th 5 min e massa de Ad de 0,5g	27,533	0,246	62,301
Th 30 min e massa de Ad de 0,5g	35,861	0,313	81,153
Th 60 min e massa de Ad de 0,5g	37,477	0,326	84,810
Th 5 min e massa de Ad de 10,0g	43,008	0,3705	100,000
Th 30 min e massa de Ad de 10,0g	26,414	0,237	61,416
Th 60 min e massa de Ad de 10,0g	26,787	0,24	62,283
Th 5 min e massa de Ad de 10,0g	27,657	0,247	64,306

With the data obtained (Table 8) it was possible to observe that the adsorption process using 0.5 and 10 g is efficient up to approximately 5 min of contact (Figure 7). After 5 min, the treatment with a mass equal to 10 g tends to remain constant, while the concentration in the sample carried out with the treatment containing mass equal to 0.5 g, after 5 min, increases again.
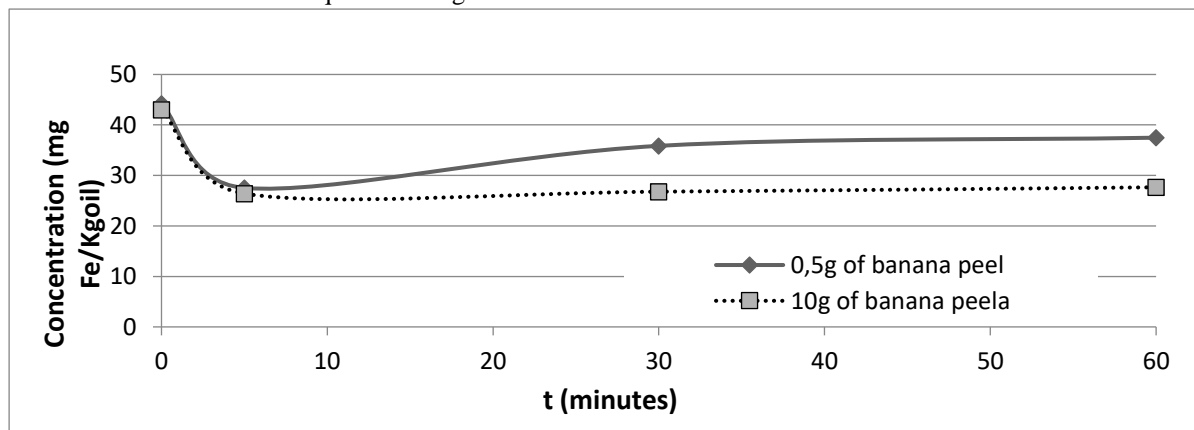

Fig.7: Residual concentration in mg Fe / Kg oil over time for a sample with 0.5g and 10g of husk, separated by vacuum filtration.

V. CONCLUSION

As stated by Jawahir and Bradley, 2016 the practices of the 6R concept contribute to reducing the impacts generated by the incorrect disposal of this waste. The use of organic adsorbents is one of the tools to achieve this goal.

The separation processes by vacuum filtration and centrifugation were efficient in all tests, managing to separate the iron ions from the oil by chemical adsorption and by the action of the centrifugal force, respectively.

The results obtained by the solid-fluid separation process by centrifugation were influenced by the centripetal force caused by the separation of the ions by dragging, but the process had an efficiency between 33.43 to 94.53% of removal of iron ions from the oil depending on the treatment time. .

The use of Musa paradisiacal banana peels for the Fe ions adsorption, using 10 g of Ad and separation performed by vacuum filtration obtained an efficiency of 38.584% during the first 5 mins of contact between the materials.

Due to the adsorption kinetics, the oil treatment using 0.5 g of peel was efficient in the first 5 min, after that time, the Fe concentration remained constant.

ACKNOWLEDGEMENTS

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