IAPE '19, Oxford, United Kingdom
ISBN: 978-1-912532-05-6

Bio-oil from Digested Sewage Sludge Pyrolysis Blended with Diesel as Alternative Fuel for Thermoelectric Power Stations

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ABSTRACT

Thermoelectric power stations (TPS), as most of the world's energy matrix, still strongly depends on fossil resources. By that, advances in technology have been trying to bring alternative solutions by exploiting waste materials to produce energy, aiming for gradual substitution or co-joint uses. Sewage sludge (SS) gained the attention of world-scale scientists mainly by its constitution, which carries considerable amounts of oil that can be recovered by current and well-diffused processes such as pyrolysis. After the discovering of fuel generation possibility from SS, pyrolysis has been implemented in many different forms to optimize the bio-oil obtaining and, in this study, as we aim for medium or long-term partial substitution of diesel in TPS's, we developed a 1:1 blend of diesel with bio-oil produced by slowly pyrolyzing digested SS, collected in two different seasons (dry and rainy), determining its physiochemical and chemical fuel properties. We were able to maximumly obtain 4.208 and 7.352 g of bio-oil from each digested SS samples (40 g) and their blending with diesel have enhanced density, calorific value and pH in 6.64, 21.85 and 5.83%, respectively. Many fuel-related compounds were found in the GC/MS analysis. Further transesterification studies and deeper analyses are necessary for optimization and fitting in biodiesel standard regulations.

Keywords
Waste exploitation, applied energy, biofuel, environment.

1. INTRODUCTION

In Brazil reality, 2 million cubic meters of diesel are consumed by year to maintain about 2000 thermoelectric power stations, generating a quarter of the energy distributed throughout the country, with estimated installed-power of 37.000.000 kW [1]. This dependence of fossil fuels, not only in Brazil, has been trigging the ongoing research fields on renewable energy and the biodiesel production has been now exploring waste materials feedstocks such as digested sewage sludge (SS) [2].

Digested SS is produced from primary and secondary sedimentation processes in most of the Residual Water Treatment Plants (RWTP), with high quantities of organic matter, minerals, and water, representing a significant fraction of the not-recovered energy within the treatment plant [3]. Conventionally, sludge amount depends on the characteristics of the effluent and treatment process applied [4], also regulating the oil presence in its constitution [5].

The possibility of fuel from digested SS, mostly due to the low nitrogen and sulfur content [6], has unleashed research lines using thermo-chemical conversion processes for its acquisition, whereas the most commonly described is pyrolysis [7]. The bio-oil obtained from sewage sludge pyrolysis is a dark-brown liquid with a strong and specific smell, and its composition has been described as potentially not harmful when relating to greenhouse effects [8].

Thereby, mixing diesel and bio-oil as fuel supply in thermoelectric power stations might help to decrease fossil fuels dependence, also promoting waste reuse, production costs reduction and diminishing emissions of sulfur oxides, nitrogen oxides, carbon dioxides, methane, carbon monoxide, and particle materials [9]. Thus, this study aimed to produce, analyze and characterize bio-oil from digested sewage sludge, generated in residual water treatment plants, as a fuel supply for medium or long-term partial substitution or co-joint use with diesel oil in thermoelectric power stations.

2. MATERIAL AND METHODS

2.1. Sampling Procedures

Figure 1 shows the sewage sludge as they were received in the laboratory. Its collection in the RWTP obeyed Brazil's technical norms NBR 10.007 and NBR 10.004, that describes eligible criteria for sampling solid residues, and its classification as non-dangerous and non-inert residue, respectively [10, 11]. The material was dried in stove at 60 ºC until reached constant mass, which took approximately 10 days. After dried, both materials were crushed separately in a knife mill and homogenized in a 0.59 mm mesh.

Figure 1. Digested sewage sludge: (A) dry season and (B) rainy season

DOI: http://dx.doi.org/10.17501.....

2.2 Proximate Analyses

Moisture, volatile solids, ashes and fix carbon content analyses were conducted to characterize each digested sewage sludge as biomass samples, following what is preconized by the International American Society for Testing and Materials – ASTM, on the regulations E 871, E 872 and D 1102 [12 – 14].

2.2. Pyrolysis Procedures

Figure 2 shows a scheme of the pyrolytic system applied for bio-oil production. Approximately 40 grams of dry and rainy season sewage sludge was used in triplicate. Samples were weighted into an open-ended quartz tube (length = 75 mm, inner diameter = 2.8 mm) and fixed by loosely fitting quartz wool on both ends and placed into a 3 cm diameter and 100 cm length filament coil pyroprobe. Pyrolysis configuration was a temperature of 550 ºC, a heating rate of 10º C min^{-1}, a heating time of 120 min. Samples weights were taken before the pyrolytic process, using a balance of 0,0001 g precision. Each sample was analyzed in triplicate. The reactor operated in an inert environment, whereas the non-condensable generated gases could leak by the gas outlet and go to a treatment system consisting of three gas scrubbers. At the end of the process, the solid fraction retained inside of the quartz tube was collected, and the liquid fraction, composed by the bio-oil and aqueous fraction, was recovered through the separating funnel.

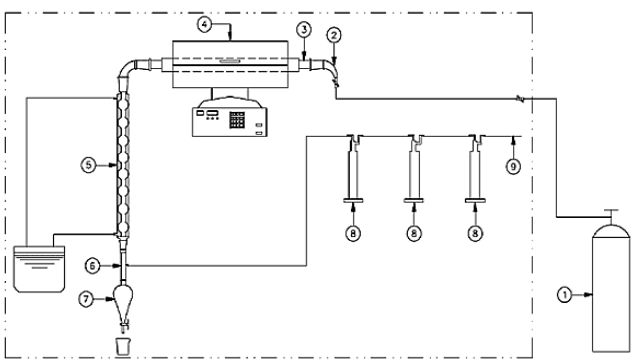

Figure 2. Scheme of the pyrolytic system where (1) nitrogen cylinder; (2) inert gas inlet; (3) quartz reactor tube; (4) oven; (5) condenser; (6) gas outlet connection; (7) separating funnel; (8) gas scrubbers; and (9) gases outlet

2.3. Physiochemical Characteristics

Analyses of density, calorific value and pH were conducted using samples of sewage sludge bio-oil after pyrolysis and then of the 1:1 blend with diesel oil. All these analyses were made in triplicate. Density was determined by a model DMA 35-EX digital densimeter. Calorific value was measured using a calorimetric bomb coupled to a model C200 calorimeter, composed by a vase thermally isolated (adiabatic), filled up with 2 L of deionized water, endowed with an ignition device through electric current, and 1,0 g sample of both bio-oils and the 1:1 blend with diesel, placed into a porcelain crucible inside a metallic recipient (calorimetric bomb). The metallic recipient was tightly closed and pressurized under 30 bars, in oxygen excess. An MB 10 model pH reader with combined electrodes (Ag/AgCl) and relative precision of 0,01% (Fe) was used to measure pH value. For that, 1,0 g sample bio-oil and the blend of bio-oil and diesel was suspended, separately, in 1:5 (m/v) deionized water prepared into a 10 mL becker.

2.4. GC/MS of the Bio-oil

40 µL of derivative agent MSTFA (N-Methyl-N-(trimethylsilyl)trifluoroacetamide) was added to 20 mg of both rainy and dry season bio-oil, separately and non-sequentially, and left in a 40 ºC water bath during 15 minutes, then each diluted in 1 mL of dichloromethane. CG/MS was conducted on a model Varian 2200 chromatograph, using 99.9999% helium as carrier gas, column flow of 1 mL min^{-1}, injector and detector temperature of 300º C, Split type injection, 20:1 ratio, capillary column of 60 m x 0.25 mm x 0.25 µm (VF – 5 ms) (5% phenyl and 95% methyl silicon). The temperature schedule was: initial temperature of 40 ºC, ramping to 100 ºC in a 5 minutes isotherm, sequentially ramping to 250 ºC in a 5 ºC min^{-1} rate, and then to 300 ºC in a 20 ºC min^{-1} rate, both last within a 30 minutes isotherm.

3. RESULTS AND DISCUSSION

3.1 Proximate analyses

Data of moisture, volatile solids, ashes and fixed carbon contents (mean ± SD) are presented in Figure 3. There was a statistical difference ($p = 0.0149$) between the values of humidity and volatile solids from each season samples, which are directly affected by the water content, as it can adjust the behavior of the chemical compounds into the biomass [15]. These four parameters preliminarily indicate if the waste biomass has the potential of producing bio-oil, mainly by the conversion of carbonic compounds in the presence of water and organic matter [16].

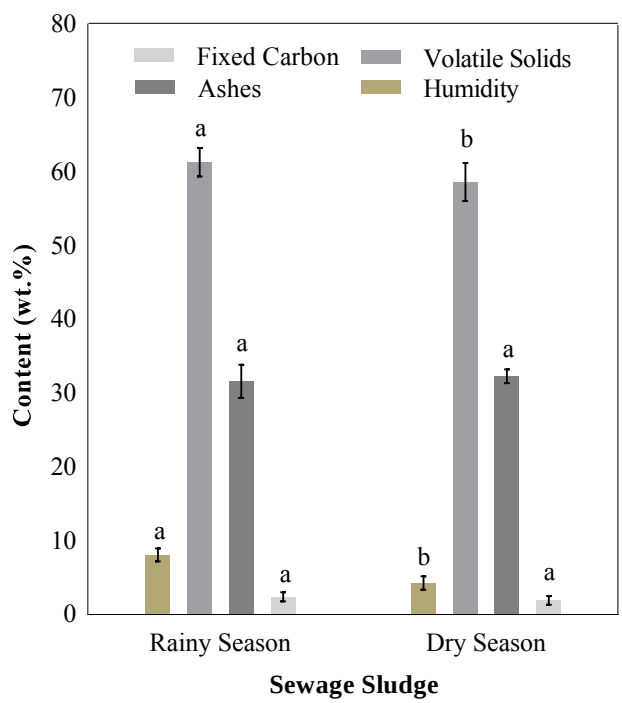

Figure 3. Average values from the proximate analyses conducted on both rainy and dry season sewage sludge samples. Same lowercase letter upon the bar of each parameter indicates no statistical difference.

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The amount of water present in the digested sewage sludge causes influence on the combustion processes [15]. If present in high content, the water can drive to the need of pre-drying the biomass, which is a procedure with high energetic waste, and should be avoided [17]. The water also is responsible for reducing biomass calorific power, i.e., whilst spending time on drying the matter, more consumption of energy is necessary, which determine the amount of energy effectively available versus the free-water content [16].

Sewage sludge is rich in volatile material, as can be noticed on the values found out in this study (58.10 and 61.19 wt.%). Conversion of biomass with high contents of volatile material is a key for bio-oil yield increase [15, 16], it also eases ignition and produces more large flames, therefore, as higher the quantity of volatile solids is, higher will be the yield of bio-oil or gas onto pyrolysis, tending to have high calorific value too [18].

In pyrolytic processes, volatile solids have great importance because of their role on organic matter constitution, enabling to produce sub-products rich in energetic-compounds such as hydrocarbons (alkanes and alkenes), carboxylic acids and alcohols of high molecular weight [15, 17, 19]. Another important parameter is the ashes content, which depends on the chemical characteristics of the sewage, being mostly related to human population habits, tending to differ around the world [20]. Same goes for fix carbon, responsible for maintaining the combustion stable, therefore, as higher the carbon concentration is, higher will be the material's calorific value [20, 21].

3.2 Yield and physiochemical characteristics

Tables 1 and 2 present bio-oil yield values of both rainy and dry season sewage sludge samples, and their physiochemical characterization, respectively. Bio-oil is mostly a mixture of hundreds of organic compounds, fuel and non-fuel related, with different chemical proprieties [15]. The amount of liquid fraction obtained in pyrolytic processes is mainly related to the feedstock type [15]. In the case of sewage sludge, other factors such as sewage treatment type and the pyrolysis heating time and heating rate also may influence [22].

On the other hand, pyrolysis processes can also be affected by the presence of inorganic compounds (mainly metallics such as Ca, K, Na, etc.) [23]. Normally, these elements are responsible for accelerating dehydration and carbonization reactions during primary and secondary reactions, resulting in reductions on the liquid fraction sub-product (mainly hydrocarbons) and increase gas and solid materials production [24, 25].

Our sampling conditions under pyrolytic effect yielded 18.38 wt.% for rainy season sewage sludge and 10.52 wt.% for dry season of bio-oil. The literature reports similar yield values, despite the sampling conditions [16, 26, 27, 28].

Table 1. Average values and variance coefficient for bio-oil yield†

Samples	Bio-oil (wt.%)
Rainy Season	18.38 ± 0.34 a
Dry Season	10.52 ± 0.26 b
VC%	2.73

† Values followed by the same character in the column does not statistically differ between themselves by the T-test ($p < 0.05$).

Table 2. Physicochemical characteristics of the bio-oils and their 1:1 blend with diesel

Sample	Density (kg m^{-3})	Calorific Value (MJ kg^{-1})	pH -
Rainy Season	971,2	32.36	8.35
Dry Season	958,7	31.02	8.11
RS + Diesel	943,6	41.41	7.89
DS + Diesel	910,7	39.57	7.71
vc (%)	2.76	14.1	3.45

Legend: RS - Rainy season; DS - Dry season; vc (%) - variation coefficient.

Density is important to indicate oil quality, being susceptible to changes because of impurities or adulteration on the oil original composition [29, 30]. Common commercial diesel density values go from 820 to 850 kg m^{-3} [31], which are close to the values found for sewage sludge bio-oil. Here we must take into consideration that this comparison stands for crude bio-oil, i.e., after the necessary procedures of refining, cleaning, and filtering, they may or may not get even closer.

Calorific value is the most important parameter to be taken into consideration, as it stands for the energetic potential during the combustion of the fuel [15, 19]. This parameter might variate accordingly to biomass type, chemical composition and moisture content [19]. Some of the worldwide standards do not define a pattern to biodiesel calorific value as Brazil's and EU's regulations do and this study's results are close to these required values, whereas this parameter indicates the potential of the biodiesel as fuel and its improvement has been studied with diesel blends, in order to reduce pollutant emissions and costs of product, as well as slowly inserting a more environmental-friendly type of fuel in market [32, 33].

The main concern about pH is based on it not being too acid (values below 4) [20, 34], whereas it may compromise the metallic structures of the motors [28, 35]. This parameter can be highly influenced by its acid compounds and storage conditions, contrastingly, amines content confers to it more alkaline characteristics [27, 36].

Table 3 shows the improvement caused by the 1:1 blend with diesel for each sampling type. It is noticeable an enhancement of the physicochemical proprieties, enabling the partial substitution, which can reduce both financial spending and pollutant emissions, offering an environmental gain and contributing to greenhouse effects reduction.

Table 3. Enhancement percentage of the bio-oil 1:1 blend with diesel

Blend 1:1	Enhancement Percentage		
	Density	Calorific Value	pH
RS + Diesel	2.95%	21.85%	5.83%
DS + Diesel	6.64%	21.61%	5.19%

Legend: RS - Rainy season; DS - Dry season;

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Figure 4 shows the zoomed-out chromatograms obtained during the GC/MS analysis of dry season and rainy season sewage sludge bio-oil, highlighting the peaks of each compound detected. Table 4 presents the compounds with the highest relative intensity peaks in the mass spectrum, as indicated by letter characters in the mentioned image. Analysis of the bio-oil GC/MS indicates a similar profile to petroleum, being constituted by substances of interest such as toluene, ethylbenzene, and silane, dimethyl(octadecyloxy)propyl-, mostly found in rainy season samples.

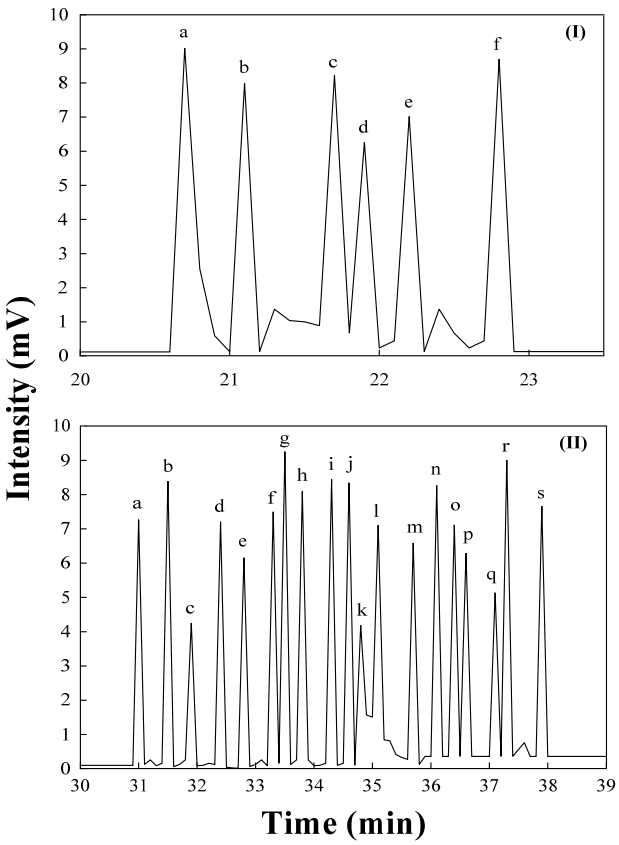

Figure 4. Zoomed-out chromatograms obtained for (I) dry season and (II) rainy season, highlighting the peaks of each compound detected

Table 4. Respective compounds in the chromatograms obtained in dry and rainy season sewage sludge bio-oil

Peak	Compounds	
	Dry Season	Rainy Season
a	Hexamethyldisiloxane	Ethyl(icosyloxy)silane
b	Trimethylsilylglycerol	(Hexadecyloxy)trimethylsilane
c	Myristyl Alcohol	Hydroxy(nonadecyl)silicon
d	1-Dodecoxy-1-ethylsilinane	Tricosane-1,15-diol, bis(O-trimethylsilyl)-
e	Chloromethyl-dimethyl-pentadecan-6-yloxysilane	Trimethyl(nonadecoxy)silane
f	Octadecyldimethylsilanol	Trimethylsilyl (9Z)-9-hexadecenoate
g	-	chloromethyl-dimethyl-tetradecan-3-yloxysilane
h	-	4-Hexadecoxy-2-pentadecyl-1,3-dioxane
i	-	2-Trihexylsilyloxyoct-3-ene
j	-	trimethylsilyl (Z)-octadec-9-enoate
k	-	Trimethylsilyl stearate
l	-	Trimethylsilyl heptadecanoate
m	-	(dichloromethyl) (heptadecyloxy) dimethylsilane
n	-	Cholest-2-ene, (5alpha)-
o	-	[(6,6-Dimethyl-3-pentyl-9-([(tripropylsilyl)oxy]methyl)-6a,7,8,10a-tetrahydro-6H-benzo[c]chromen-1-yl)oxy](tripropyl)silane
p	-	Toluene
q	-	Ethylbenzene
r	-	Trimethyl(pyridin-3-yloxy)silane
s	-	Trimethylsilyl laurate

4. CONCLUSIONS

This and other studies have indicated sewage sludge as a feasible biomass to obtain bio-oil in thermochemical processes. 1:1 blending of diesel with bio-oil obtained in digested sewage sludge pyrolysis has promising future for medium or long-term partial substitution, or co-joint use, in thermoelectrical power plants, as it shows preliminarily satisfactory density, calorific and pH values. It is recommended to develop further studies of transesterification and deeper analyses of other physical and chemical properties, also suggesting the execution of engine tests to study its performance and pollutant emission potential.

5. ACKNOWLEDGMENTS

We would like to thank LEDBIO for providing support to developing this study.

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