COMPARATIVE STUDY ON THE DIFFERENT EXTRACTION PROCESSES FOR *Kigeliaafricana* BIO-OIL

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Abstract

Kigeliaafricana bio-oil is potential feedstock for the production of biodiesel. This bio-oil could be efficiently extracted so that the feedstock cost could be reduced. A comparative study on the extraction process have been studied using Soxhlet extraction, aqueous acid extraction, two phase solvent extraction and ultrasonic assisted extraction process. The yield of oil and acid value was the two parameters focused. The highest yield of oil was observed for the Soxhlet extraction method with 45.1% and the lowest acid value was observed for the two-phase solvent extraction method as 4 mg KOH. The physiochemical properties of the oil were studied and for all the methodologies it was found to be within ASTM standard limits.

INTRODUCTION

The two major issues in the recent years are the shortage of energy and the concern for the environment. A pollution free atmosphere is the need of the hour. The major conventional methodologies adapted for the generation of energy are fossil fuels, hydro-energy, wind energy, tidal energy and few more [1]. Among the above-mentioned techniques other than fossil fuels other technologies are renewable energy sources. In the transportation sector still 80 % of the energy is dependant only on petroleum derived fuels namely gasoline, diesel and natural gas [2]. The emission of greenhouse gases is at alarming levels and many environmentalists are working on alternative fuels with minimum emission of gases. The other drawback in using fossil fuels is that the source available in current scenario is highly limited[3]. A prediction had been made stating that these fossil fuels would be available only for two to three more decades [4]. There is a startling situation for energy deficit currently to opt for new viable budget and environmentally friendly biofuel[5]. The research on biofuels is not brand new. This had been performed for past few decades and the first generation of biofuels had also been successfully accomplished. But the drawback is it may affect the food security and thereby alternative feasible sources of biofuels had been into recent research[6]. The non-edible resource could be categorized under second generation of biofuels.

Biomass could be categorized as a potential feedstock for the production of clean carbon free biofuel. It is majorly composed of complex bioactive compounds. Biodiesel could be produced from the feedstock with high lipid content [7].The promisingraw material should possess oil rich in triglycerides. There are many challenges in biodiesel production from these second-generation feedstocks. One of the major challenges could be the composition of the fatty acid types present in the oil. This varies from one biomass to other. The other significant thing to be considered is the solvents used for the extraction of lipid from the biomass[8]. This would alter the composition of the extracted oil. Solvents based on their polarity are chosen for the extrusion of lipids[9]. Mostly non polar and intermediate polar solvents are chosen for the work. Oil being non polar in nature it would be extracted efficiently with similar non polar solvents. Lipid extraction is one of the significant steps in the biofuel production. Selection of proper feedstock with high lipid content would be have to be chosen wisely [10].

Lipid extraction from the feedstock is an important step in the production of biodiesel. There are many conventional methods been followed for the extraction of oil namely solvent extrusion, soxhlet extraction, ultra-sonication, supercritical fluid extraction, microwave assisted extraction and many more methods.However, due to the high efficiency and low selectivity of these methods, they can result in the extraction of pigments, proteins, and carbohydrates [11], which are not suitable for biodiesel production and can compromise analytical results in quantifying lipid content.

Kigeliaafricana commonly called as sausage tree is a pantropically available in different parts of Asia and Africa. This tree bears a fruit in sausage shape and it has many medicinal effects [12]. In countries like India, it is used as an ornamental tree as it has attractive red coloured flowers. The seeds from these fruits are non- edible and also have not been used as the feedstock earlier for the biodiesel production.

The current work has been focused on extraction of lipid from *Kigeliaafricana* seed biomass. A comparative study on the methods adapted have to be made on the fatty acid profile of the extracted oil and the physiochemical properties of the extracted oil. A kinetic study has also been made.

MATERIALS AND METHODS

*Kigeliaafricana*fruits have been collected from the trees found in the campus of Anna University, Chennai. The seeds were segregated and shade dried. This ws used as the biomass for the extraction of lipid.

Different solvents namely n-hexane, petroleum ether, chloroform, methanol, acetone and sulphuric acid were of analytical grade with a high purity of 99.9 %. These chemicals were purchased from Merck India Pvt. Ltd.

METHODOLOGY

The extraction methods used in the methods used are Soxhlet method, aqueous acid extraction, two phase solvent extraction, ultrasonication methods and direct extrusion.

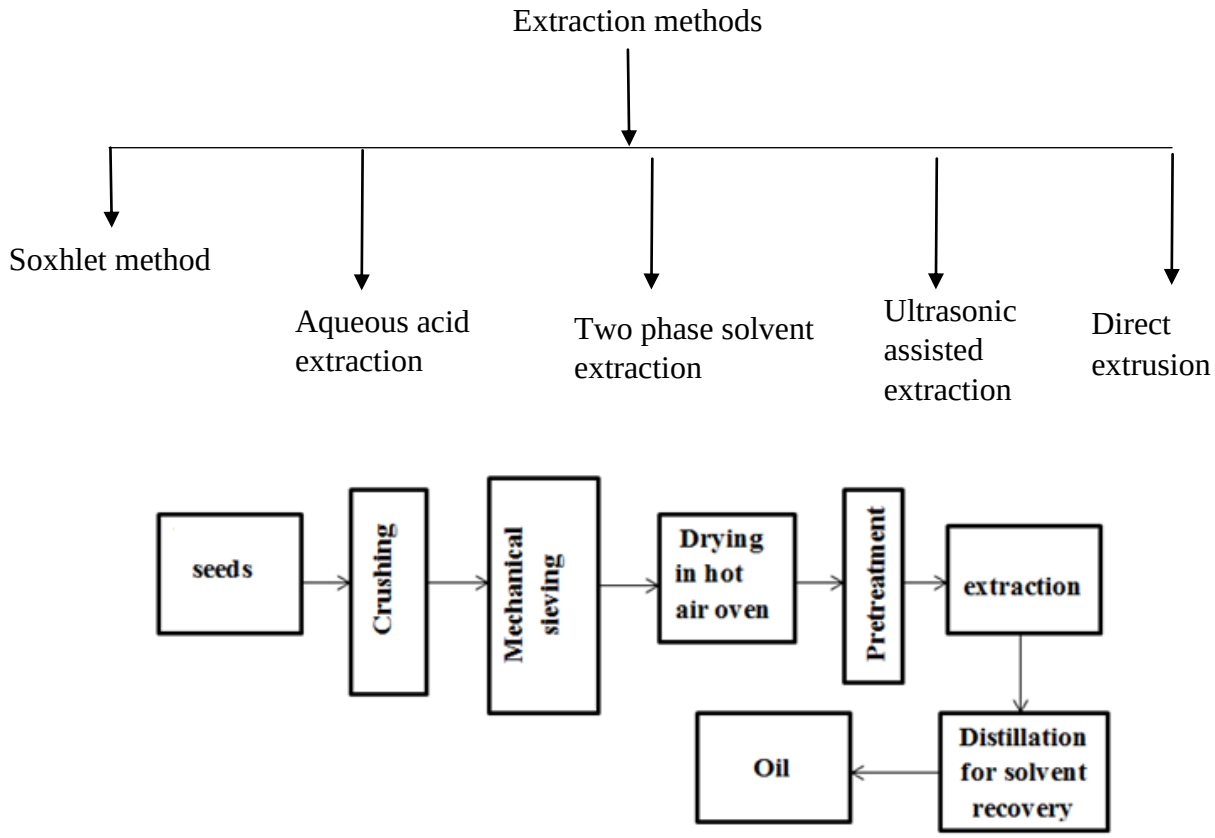

Figure 1 Flowsheet on extraction of bio-oil from *Kigeliaafricana* biomass

SOXHLET EXTRACTION METHOD

Soxhlet extractor was utilized for the extraction of oil from the biomass. n-Hexane solvent in the ratio of 1:20 in the two-neck flask. This was attached to the Soxhlet extractor fixed with a condenser. Water is refluxed continuously in and out of the condenser. The biomass was taken in the thimble and placed inside the extractor. The extraction was carried out at the boiling point of the solvent which is 65 ºC. The solvent got vaporised at its boiling point. This

vapour gets condensed and comes in contact with the biomass.This helps in indirect contact extraction of oil from the biomass. Both oil mixed with n-hexane rolls back to the flask. The solvent is recovered by distillation process and used for next cycle of experiments.

AQUEOUS ACID EXTRACTION

Aqueous sulphuric acid solutions of varied concentrations say 5%-25 % (with 5 % incremental) were prepared. The seed powders were mixed with n-hexane and prepared sulphuric acid mixture in flat bottomed flask and connected to a reflux condenser. The FB flask was placed inside a water bath positioned over a hot magnetic plate. The working temperature was maintained constant throughout the process. Continuous stirring of the mixture was performed using a magnetic pellet. Extraction of oil from the seed powder happens when the process in extended for few hours. After the process completion, powder is filtered from the oil- solvent mixture using a Whatman filter paper. The acid value of the extract was calculated. The difference between the final residue measured and initial known weight of the seed powder would be quantified as the oil content. Optimization of oil extraction was performed at different time intervals and change in system temperature.

TWO PHASE SOLVENT EXTRACTION (TPSE)

This method of extraction was performed using mixture of polar and non-polar solvents. the experimental methodology was same as the aqueous acid extraction. N-hexane and aqueous methanol was taken in the ratio of 60:40 and the yield of oil was calculated. Non polar solvent was chosen for the extraction of lipids from the biomass as they are highly non-polar in nature. The oil has free fatty acids and other impurities has been extracted using the polar solvent methanol.

After the extraction the filtrate is poured in separating funnel until the phases are separated. Two distinct layers were observed. The top layer was oil with n-hexane and the bottom layer was methanol with water, FFA and other residues. The oil yield and FFA was analyzed.

ULTRASONIC ASSISTED EXTRACTION

The extraction of oil was performed using ultrasonic waves assistance. For this method a probe ultrasonicator was used (Make: Sonics Vibra cell). 25 hz frequency of ultrasonic waves and the power of 500 watts were the configuration of the used probe sonicator. The biomass with the solvent was taken in the beaker and it was kept in the ice bath. This whole set-up was kept inside the probe

sonicator and the ultrasonic waves subjected with an on and off cycle of 10 seconds. The ultrasonic waves subjected created a cavitation effect in the mixture and this in turn helped in the fracturing of outer walls of the biomass. Pores have been created resulting in better oozing of oil from the seed powder. After a short period of time, the solution was undisturbed and it was filtered. The filtrate was subjected to distillation for the recovery of solvent. The oil yield was calculated.

DIRECT EXTRUSION METHOD

10 g seed powder was taken in the conical flask. The different solvents were taken in the volumetric ratio of 1:10. The contents in the flask were shaken continuously at regular speed using temperature controlled orbital shaker. The process is continued at constant atmosphere for few hours. After the extraction the contents are transferred to the Buchner funnel and the vacuum filtration is performed. The solvent is subjected to rotary evaporation and the extracted oil is analysed.

RESULTS AND DISCUSSION

COMPARATIVE BIO OIL YIELD

The oil yield from different methodologies were obtained and they were consolidated in the figure 1. It could be observed that Soxhlet extraction method has reported the highest yield of oil with 45.1 %. The lowest yield of oil was noted for the direct extraction method.

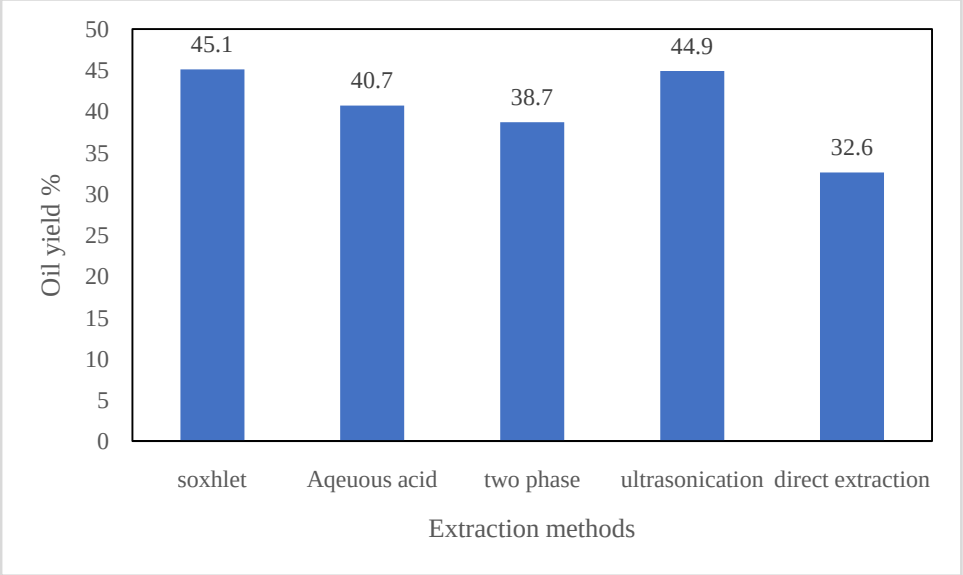

Figure 2 Comparative analysis on oil yield % from different extraction methods

Though n-hexane was used as the solvent for all the extraction methods it was used in different combinations. For Soxhlet, ultrasonication and direct extraction n-hexane was taken in the ratio of 1: 20 (biomass to solvent). For aqueous acid extraction method, 20% of sulphuric acid was mixed with n-hexane. For two phase solvent extraction method, n-hexane and aqueous methanol were taken in the 80:20 ratio.

The next parameter to be analysed in the bio-oil is the acid value. For the extracted bio-oil to be utilized as the feedstock for the production of biodiesel the acid value has to be below 5 mgKOH. If the value is high, two step transesterification had to be performed. The acid value was found for each extracted bio-oil and it is represented in figure 2.

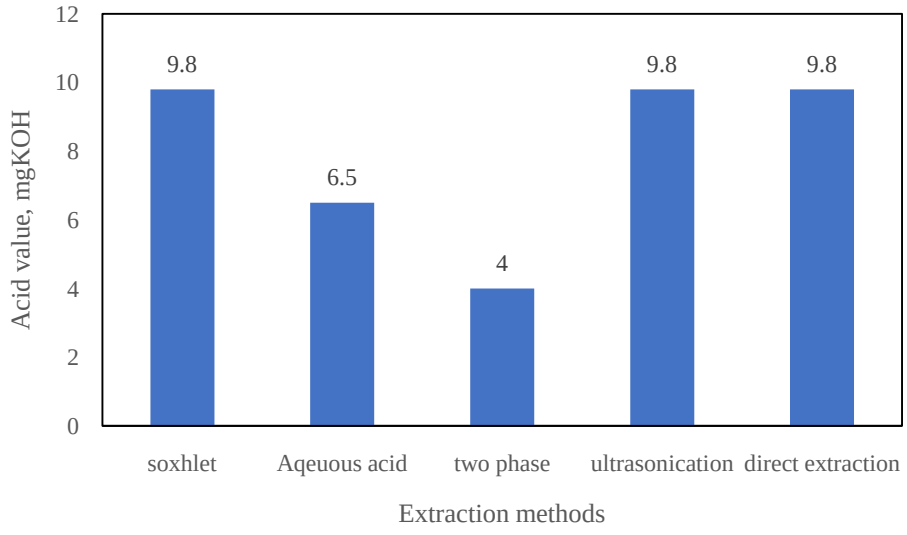

Figure 3 Comparative analysis in acid value mg KOH from different extraction methods

From the figure 3, it could be observed that the acid value below 5 mg KOH has been observed only for two phase solvent extraction method. In TPSE method, the polar components namely FFA and other impurities were extracted by the polar solvent aqueous methanol. The oil was extracted by non-polar solvent n-hexane. The extracted oil only through this methodology could be subjected for direct transesterification process. The oil from other extraction methodologies requires acid pre-treatment.

Effect of time on extraction

The time of extraction for each method was compared with the yield of oil in figure 4. Other than ultrasonication method all the other extraction methods were time consuming process. The best yield of oil was obtained only after 5 hours for the other methods of extraction

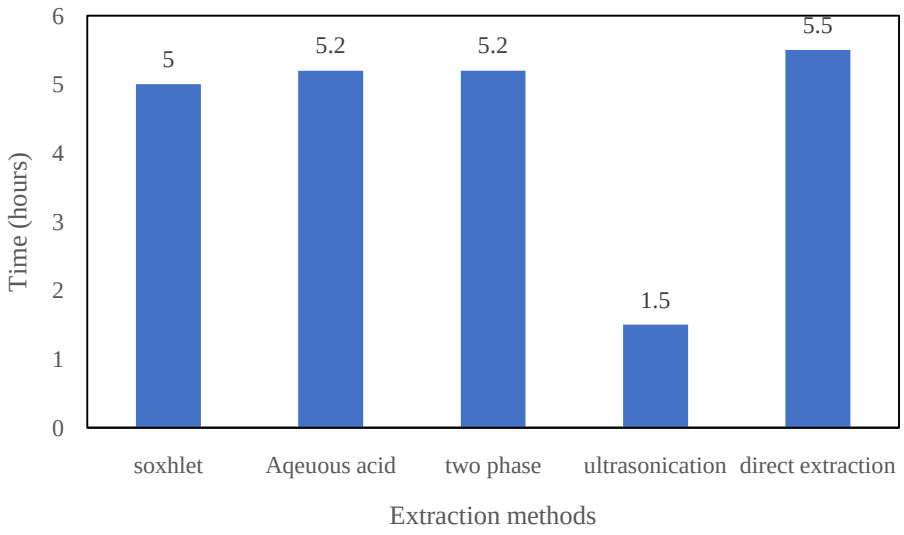

Figure 4 Comparative analysis on extraction time from different extraction methods

PHYSIO-CHEMICAL PROPERTIES OF EXTRACTED OIL

The physical and chemical properties of the extracted oil from all the methodologies were analysed and they are reported in Table 1.

Property	Soxhlet	Aqueous acid	TPSE	Ultrasonication	Direct extraction	ASTM methods
Colour (Lovibond no)	24	17	17	13	24	--
Odour	Repulsive	Repulsive	Repulsive	Repulsive	Repulsive	--
Specific gravity at 25°C	0.927	0.919	0.91	0.92	0.92	ASTM D1840 method
Kinematic viscosity(cst)	41.1	41.0	40.9	41.1	41.2	ASTM D445 method
Iodine value(gI_2/100g)	106.5	68.3	68.4	106.7	106.6	
Saponification value	205.2	203.1	204.1	204.6	205.2	
Cetane number	57.51	57.25	56.1	58.32	57.3	

	(47-65)					
Average molecular weight	861.84	861.84	861.84	861.84	861.84	

Conclusion

The comparative study on the extraction of oil have been performed. The best yield of oil was observed for the Soxhlet method of extraction and the lowest yield for TOSE method. On contradiction the acid value was observed to be lowest as 4 mg KOH for TPSE method. The yield of oil was not very less for TPSE method. The acid value is the important parameter to be considered than the yield for the biodiesel production. Considering this fact, it could be concluded that TPSE methodology could be comparatively the best method for the extraction of oil. The physical properties for the extracted oil from all the methodologies has been almost similar. No huge variations were observed. As per the ASTM standards the properties are within the limits hence this oil could be utilised for the transesterification process for the production of biodiesel.

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