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Molinillo Seed (*Hura crepitans*) Subjected to Different Drying Techniques and the Effects on the Bio-oil Quality Potentials

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Abstract
This study assessed the impact of different drying processes on the bio-oil yield and physicochemical parameters of molinillo seed (*Hura crepitans*), an underused non-edible oilseed with promise for renewable energy applications. Using a complete randomized design with three separate trials, seeds were dried by oven, vacuum, solar and sun, with an undried sample that served as control. Whilst solar and sun drying occurred in ambient settings, oven and vacuum drying were carried out at 40, 50, 60, and 70 °C respectively. Soxhlet extraction with n-hexane was used to extract bio-oil, and the oil yield was calculated. Using conventional AOAC and ASTM procedures, as well as some modified methods, the extracted oils were examined for acid value, saponification value, iodine value, peroxide value, specific gravity, and flash point. Analysis of variance was used to statistically examine the data at an accuracy level of 95%. The findings demonstrated significantly increased oil yield with the oven and vacuum drying, where vacuum drying generally yielded more stable oil, in comparison to solar and sun drying. Oven drying at 40°C yielded the maximum oil output (49.70%), while sun-dried sample yielded the lowest (36.97%). Oils with reduced acid and peroxide values, and improved oxidative stability due to reduced moisture were also, consistently obtained by the vacuum drying. Iodine values (83.0–87.5 g I_2/100 g) and saponification values (193.55–200.30 mg KOH/g) showed suitability of the oil for industrial uses, such as, the manufacturing of soap and biodiesel.

Keywords: Molinillo seeds, bio-oil yield, renewable energy, drying methods, potential qualities

1. Introduction

The increasing global demand for energy, coupled with the depletion of fossil fuel reserves and environmental concerns, has led to a surge in the search for alternative renewable energy sources (Kenenu and Marchetti, 2017). Vegetable oil which is a renewable raw material is basically of household and commercial relevance globally, depending on its physicochemical properties (Akpe and Oguntunde, 2019). In Nigeria, for instance, the commonest vegetable oils serving the dual purposes are those of groundnut, oil palm and soyabean (Ayodele et al., 2017; Esonye et al., 2019). However, these oils are edible, and as such are confronted with the challenge of diverting supposed food to biofuel, which may not only cause food scarcity in an undeveloped economy, but can also trigger a somewhat nutritional and ethical uproar (Demirbas et at., 2016).

Non-edible seed oils which are toxic or non-toxic biodegradable substances, are these days, commonly finding their applications in the production of industrial commodities, such as biodiesel, soaps, paints, bio-lubricants, cosmetics, etc. (Islam et at., 2018; Singh et at., 2019; Ndukwu and Onyeoziri, 2020; Nelson, 2022). Some of the common non-edible oilseeds are Rubber seed, Trichosanthes cucumerina, Jatropha curcas, Thevetia nerifolia and *Hura crepitans*, (Ominowa et al., 2024).

Molinillo seed (*Hura crepitans*), is a lignocellulosic biomass emanating from a continuous fresh leafy tropical plant species, that was originally, linked to America (Gbadamosi and Osungbade, 2017; Onwe and Bamgboye, 2021). The mature fruit is in form of a pumpkin fruit, but when dry, explodes and releases disc-like seeds of averagely 20mm thick, well capsulated on the average of 15 seeds which are separated from each other by septa in the fruit pod approximating 40 mm high and 60 -70 mm wide (Gbadamosi and Osungbade, 2017; Onwe and Bamgboye, 2021).

Oil is normally retrieved from feedstocks, after certain pre-operations, have been conducted, such as, de-husking, cleaning, moisture reduction and grinding (Ndukwu and Onyeoziri, 2020). However, seed moisture reduction to a certain level that decreases microbial activities, minimizes fat breakdown, prolongs shelf life, preserves the oil-bearing cells as well as enhances oil quality and yield, requires effective drying (Alara et al., 2017; Iqbal et al., 2019; Mitrović et al., 2020), which can be, significantly influenced by the drying method employed prior to oil extraction (Wanyo et al., 2015; Qu et al., 2016; Ndukwu and Onyeoziri, 2020). Studies have also, shown that lowering of moisture content in agricultural and medicinal plant materials is normally done by convective drying (Ndukwu et al., 2017); which requires a precalculated release of heat flow in a drying component within a specified period (Ndukwu et al., 2018a). Perhaps, the inclusion and employment of other form of drying such as, vacuum drying can surmount the limitations of convective drying on heat sensitive compounds (Zhang et al., 2018).

The influence of drying methods on the yield and quality of oil from oil plant materials have been examined by a number of researchers. Study by Silva et al. (2020) revealed that drying aided the retention of low acidity level in Macauba fruits. The influence of drying techniques on the quality and yield of oil from avocado pear has been investigated, of which a higher yield from hot air against open air drying was revealed (Chimsook and Assawarachan, 2017). Zhang et al. (2018) had as well studied the effect of drying techniques on the quality of flaxseed, and revealed that more yields were witnessed from the microwave and freeze drying over the conventional techniques. It was also noticed that drying temperature had no effect on both the peroxide value and the percentage free fatty acid of sunflower seed oil as reported by Ndukwu and Onyeoziri (2020). The impact of drying on the physical properties and stability of encapsulated pumpkin was also investigated, which revealed that spray drying provided the highest yield at 130ºC, at which an increased temperature, provided more oil yield (Ogrodowska et al., 2017). However, a thorough search through the literature has revealed shortage of study on the impact of drying techniques on the yield and quality of oil extracted from molinillo seed.

Molinillo trees are widely spread all over many arid and semi-arid countries, functioning as ornamental or shading plants (Onwe and Bamgboye, 2021). Despite the characteristic high oil content of its seeds, ranging from 36 to 72% (Omilakin et al., 2020; Owhor et al., 2021), they have been so much underutilized, as they have not been traced to any known consumption or economic value (Giwa et al., 2016). They also, readily grow on non-arable or waste lands, and can easily be domesticated under well cultural practices at zero interference on food productions, with the seeds easily harvested and utilized almost all year round (Amine et al., 2018; Owhor et al., 2021). Considering the fact that, the seed is highly toxic, and thus, not fit for animal or human ingestion (Vassallo et al., 2020), it can however, be tapped for industrial applications. Moreso, since moisture reduction is very vital in enhancing bio-oil extraction yield, it is necessary to examine different drying techniques to identify the most desired outcomes. Thus, evaluation of the effect of drying methods on the extractable bio-oil of molinillo seed becomes very necessary.

This study aims at subjecting molinillo seed to different drying methods to determine the effects on the extracted bio-oil quality potentials. The findings of this study will provide valuable insights on the best drying techniques to enhance both quantitative and qualitative extraction of molinillo seed oil. The information is vital now that, the quest for alternative renewable sources of energy is on the increase globally (Dinçer, 2025).

2.0 Materials and methods

2.1 Materials

Mature pods of molinillo were collected from the premises of University of Nigeria, Nsukka, Enugu state, and manually opened. The seeds of premium quality were selected and dehulled with hammer for the study. Reagents of analytical grade used in this research are: N-haxane, hydrochloric acid, potassium hydroxide, iodine crystals, potassium dichromate, sulphuric acid, potassium iodide. The different dryers utilized in drying of the seed samples were drying tray,

2.2 Design of experiment

The experiment was designed using a completely randomized design (CRD) to evaluate the impact of various drying methods on the bio-oil yield and quality potentials of molinillo seed (*Hura* crepitans),

Two main factors were considered in this study and they include: drying method (which comprises, oven drying, vacuum drying, solar drying sun drying); and drying temperature. For the controlled drying methods (oven and vacuum drying), temperature was varied at four levels (40, 50, 60, and 70 °C). On the other hand, solar and sun drying were considered as single-level drying treatments as they were performed under natural ambient conditions.

In oven and vacuum drying, the interaction of drying method and temperature formed a 2 x 4 factorial arrangement, resulting in eight treatment combinations. Moreso, solar drying, sun drying and undried sample, serving as control were included to allow a broader comparison across drying conditions.

The samples were randomly assigned to the various drying processes in order to ensure reliability and minimize experimental bias; and a total of thirty-three experimental runs were produced by performing each treatment in triplicate (Table 1).

The responses measured in this study bio-oil yield, as well as major physicochemical properties, such as, acid value, free fatty acid content, peroxide value, density, viscosity, saponification value and iodine value.

The experimental structure made it possible to use a one-way ANOVA to compare all drying methods. Additionally, a two-way analysis of variance (ANOVA) was used to compare the individual and combined effects of temperature and drying method for vacuum and oven drying. Statistical significance was established at 95% confidence level ($\alpha = 0.05$).

Table 1. Summary of the experimental design for all the drying methods

Treatment Code	Drying Technique	Drying Temperature (°C)	Replication
CON	Control	Zero drying	3
OD40	Oven drying	40	3
OD50	Oven drying	50	3
OD60	Oven drying	60	3
OD70	Oven drying	70	3
VD40	Vacuum drying	40	3
VD50	Vacuum drying	50	3
VD60	Vacuum drying	60	3
VD70	Vacuum drying	70	3
SD	Solar drying	Ambient	3
SUD	Sun drying	Ambient	3

2.3 Experimental procedure

2.3.1 Seed preparation

Bulk molinillo seed (*Hura crepitans*) samples were first cleaned to remove dirt, stones and other unwanted materials. A portion of the cleaned seeds was set aside as an undried control sample to serve as baseline for comparison. From the undried sample, the initial moisture content of the molinillo seeds was determined by the application of thin layer drying technique using a digital laboratory dry oven (Model-No: DHG-9101), and then, using the eq. (1):

$$Initial\ moisture\ content\ (\%\ wet\ basis) = \frac{w_i - w_f}{w_i} \times \frac{100}{1} \quad (1)$$

Where, w_i is the initial mass of sample (kg) and w_f is the mass of dry solid sample (kg). The average moisture content of the molinillo seed was determined after three replications.

Thereafter, each 250 g of the prepared sample was dried with oven drying, vacuum drying, solar drying and sun drying. Oven drying (Model-No: DHG-9101, Lifecare Medical Limited) was carried out at controlled temperatures of 40^{o}C; 50^{o}C; 60^{o}C; 70^{o}C and dried to approximately moisture contents of 9, 8, 6 and 4% (w.b) respectively at constant duration of 3hrs, 30 min. in each drying case. In the same manner, vacuum drying (GallenKamp vacuum oven, size 1) was conducted with the same temperature levels of 40^{o}C; 50^{o}C; 60^{o}C; 70^{o}C , under reduced pressure of 40 mmHg, and also dried to moisture contents of approximately 9, 7, 4 and 1% (w.b). Solar drying was performed using passive mixed mode solar dryer; whereas, sun drying was conducted under natural environmental conditions for 3 consecutive days, at average mid-day ambient temperature of about 35^{o}C, until constant weights were attained. The weight of the seed was taken hourly to evaluate the moisture content of the drying seed (Ndukwu and Onyeoziri, 2020). After drying, the seeds were dehulled manually by cracking with a small hammer. The kernels were then, milled using a traditional hand grinder (Corona).

2.3.2 Bio-oil extraction

Molinillo seed oil (MSO) was extracted from each sample using Soxhlet extractor with hexane as solvent. Solvent extraction was chosen as a result of its high extraction capacity from oilseeds (Kumar, 2017). Care was taken to maintain the same extraction conditions, to ensure that any differences observed in bio-oil yield and quality could be attributed solely to the drying treatments. The ground sample was place in the extraction chamber and the solvent heated up with the heating mantle, which allowed pure vapour to move up the Soxhlet through a channel to the thimble where the sample was kept. On reaching the condenser, the vapour condensed and returns to the sample. The process continued until the level of liquid measured up with the siphon top at which the extract ran into the boiler. The extracted mixture was then exposed to room temperature to get rid of the excess hexane from the oil. The oil yield from the different dried samples was determined from the expression of Ndukwu and Onyeoziri (2020) as:

$$\text{Oil yield (\%)} = \frac{mass\ of\ extracted\ oil}{mass\ of\ ground\ sample\ used} \times 100 \qquad (2)$$

2.3.3 Physicochemical properties of molinillo seed oil

The extracted bio-oil samples were analyzed to determine some major physicochemical properties of the oil, which include acid value, saponification value, iodine value, peroxide value, specific gravity and fire point. Standard laboratory procedures such as, the AOAC, ASTM, Kadry (2015) and Ogbu (2017) methods were followed to ensure the accuracy and reliability of the results.

2.3.3.1 determination of acid value

The acid value (AV) was evaluated using the Kadry (2015) method. That is, a blank study was developed by dissolving five grams of oil sample in 25 ml fat solvent comprising 95% ethanol:ether (1:1vol.%) was dissolved, and titrated with 0.1 N KOH by applying phenolphthalein indicator till permanent pink colour manifested. The acid value was gotten from Eq. (3) as:

$$Acid\ value = \frac{Titre\ value * Normality\ of\ KOH * 56.1}{weight\ of\ oil\ sample} \times 100 \quad \text{(mg KOH/g)} \qquad (3)$$

2.3.3.2 determination of saponification number

The AOAC 920.160 standard method (AOAC, 2000) was used to evaluate the saponification number. Two grams of oil or biodiesel sample in combination of 50cm^3 of 0.5 M Methanolic KOH was heated till the point of alkaline hydrolysis was achieved. Using ph.ph. indicator, a blank study was adapted by titrating the mixture with 0.5 M HCl till the emergence of a permanent pink colour became evident. Then, saponification value in mg KOH /g was determined using Eq. (4) as:

$$saponification\ value = \frac{Titre\ value * Normality\ of\ KOH * 56.1}{weight\ of\ sample} \quad \text{(mg KOH/g)} \qquad (4)$$

2.3.3.3 determination of iodine value

The iodine value was investigated using the Hanus method AOAC 920.158 (AOAC, 2000). Sample of 0.3g was measured into a conical flask and added 25ml of carbon tetra chloride. Iodine monochloride was also added with a pipette in a fume chamber covered with a top stopper, and the flask vehemently agitated. It was then, isolated in a dark for 2hrs. Afterwards, a solution of potassium iodide (15ml, 20%) and distilled water (120ml) was added, and the mixture titrated with 0.1N sodium-thiosulphate solutions till the yellow colour virtually vanishes. Titration continued with few drops of sodium-thiosulphate and vigorous agitation till the blue colouration completely vanished. This procedure was utilized in the preparation of a blank test, with no sample added. The iodine value was calculated from Eq. (5) as:

$$Iodine\ Value = \frac{12.69 \times N \times (V_2 - V_1)}{w} \quad \text{(gl2/100 g)} \qquad (5)$$

Where, N = normality of sodium thiosuphate, V_1 = Volume of sodium thiosuphate, V_2 = Volume of utilized sodium thiosuphate for blank, W = Mass of the sample.

2.3.3.4 determination of peroxide value

The peroxide value was examined with the method used by Ogbu (2017). 1g of the sample was placed in 250ml conical flask. 30ml glacial acetic acid/chloroform (3:2 v/v) was also added, followed by continuous agitation until homogenous solution completely emerged. Then, 1ml solution of saturated potassium iodide solution and 0.5ml starch indicator solution were also added. The resultant solution was then titrated with 0.1N sodium thiosulphate until the blue colour completely disappeared. With blank evaluation also performed, the peroxide value was computed from Eq. (6) as:

$$Peroxide\ Value = \frac{(B_2 - P_2) \times Normality\ of\ Na_2S_2O_3}{weight\ of\ bio-oil\ sample} \quad (\text{meq active O2/kg oil}) \qquad (6)$$

Where, B_2 and P_2 represent the volume of $Na_2S_2O_3$ respectively utilized for the blank and sample titration.

2.3.3.5 determination of specific gravity

The specific gravity of the oil or biodiesel sample was determined by the use of density bottle as described by Kadry (2015). 25 ml capacity clean dry density bottle was weighed. It was then, filled with water and weighed again. Thereafter, it was filled with the oil or biodiesel sample and reweighed; and the specific gravity was determined as presented in Eq. (7) as:

$$specific\ gravity = \frac{mass\ of\ oil\ or\ biodiesel}{mass\ of\ equal\ volume\ of\ water} \qquad (7)$$

2.3.3.6 determination of flash point

This was determined using the ASTM D 93-20 method. Each of the sample was filled in the flash point cup of the Pensky Martens tester up to the marked level. Then, with the power switched on to stir and heat the sample, the pilot flame was ignited and the cup was lowered intermittently using the shutter operating knob to determine the minimum temperature at which the sample got flashed, and was noted as the flash point in ˚C.

2.3.4 Statistical Analysis and Modeling of Oil Yield

A one-way Analysis of variance (ANOVA) and the post-hoc multiple comparison tests were carried out to access the impact and comparison between all drying treatments. The significance threshold was established at $\alpha = 0.05$, and statistical significance was evaluated at a 95% confidence level. Moreso, the bio-oil yield was modelled by utilizing the response surface tool of Design Expert software (version 13 Stat-Ease Inc., USA). The independent factors were the drying temperature and moisture content of drying seed, whereas, the response was the percentage bio-oil yield. The input factors were selected and the lower and upper limits set as constraints. Then, based on ANOVA results, the best model was selected and analyzed. Then, the desirability index was applied in choosing the optimal factors (Ndukwu and Onyeoziri, 2020).

3.0 Result and Discussion

3.1 Effects of Drying Conditions on Bio-oil Yield

The bio-oil yield obtained under the different drying conditions is presented in Table 2, while the overall variation among treatments is shown in Figure 1.

From Table 2, it is clear that drying conditions had a strong influence on oil yield, which varied significantly, across most of the different drying methods from 36.97 ± 0.31^g to 49.70 ± 0.26^a (%). Oven drying at 40°C clearly produced the highest yield of 36.97 ± 0.31^g (%) and was significantly better than all other treatments. This suggests that mild heating helps preserve the oil within the seed while still removing sufficient moisture to enhance extraction efficiency. Similar observations have been reported in previous studies, where controlled drying improved oil recovery by enhancing cell permeability and solvent accessibility (Ndukwu and Onyeoziri, 2020; Silva et al., 2020). Nevertheless, non-significant effects of the drying method on Citrillus lanatus seed, as was evident on the very small margin of oil yield between oven drying (1.8%,) and sun drying (0.53%) had also been reported (Idris et al. (2017).

Vacuum drying at 50°C also gave a high yield, making it the next best condition. The improved performance under vacuum can be attributed to reduced pressure, which facilitated moisture removal while minimizing oxidative degradation of oil components. This agrees with findings by Qu et al. (2016), who reported that vacuum drying preserved oil quality and improved extraction efficiency in heat-sensitive materials. At moderate temperatures (40,

Table 2. Bio-oil yield of molinillo seed under different drying conditions

Treatment	Method	Temp (°C)	Oil Yield (%)
OD40	Oven	40	49.70 ± 0.26[a]
VD50	Vacuum	50	47.05 ± 0.25[b]
VD60	Vacuum	60	44.52 ± 0.30[c]
OD50	Oven	50	43.52 ± 0.31[c]
VD40	Vacuum	40	43.50 ± 0.26[c]
OD60	Oven	60	42.16 ± 0.21[d]
OD70	Oven	70	40.66 ± 0.23[e]
VD70	Vacuum	70	40.01 ± 0.30[e]
SD	Solar	Ambient	39.26 ± 0.25[f]
CON	Control	Zero drying	37.37 ± 0.32[g]
SUD	Sun	Ambient	36.97 ± 0.31[g]

Values are expressed as mean ± standard deviation of triplicate determinations. Means with different superscripts are significantly different at $p < 0.05$.

50 and 60°C) of vacuum drying, the treatments showed similar yields, indicating a balance between moisture removal and thermal effects. However, as temperature increased beyond this range, especially above 60°C, oil yield began to decline. This trend can be linked to thermal degradation of lipids and the loss of volatile oil fractions, as reported in studies on flaxseed and other oilseeds (Zhang et al., 2018; Wanyo et al., 2015). It can also be noticed that between oven drying at 70°C and vacuum drying at 70°C, as well as between control and sun drying respectively, there were no significant differences on their oil yields.

The lowest yields were recorded for sun drying (36.97 ± 0.31[g]) (%) and the undried control sample (37.37 ± 0.32[g]) (%), and both were statistically similar. This shows that insufficient drying limits oil extraction efficiency due to the presence of residual moisture. Comparable findings by Ndukwu et al. (2018) also, noted that uncontrolled drying conditions often result in poor moisture removal and reduced product quality and quantity.

However, most oil yields from the different drying methods, other than from control and sun drying, exceeded those from previous studies on *Hura crepitans* (32.50%), flaxseed (33.5%), African oil bean seed (35.7%), Pycnanthus angollensis (34.00%), palm kernel oil (28%), but lower than that of peanut (50%) (Akpe and Umo, 2019; Zhang et at., 2018; Esonye et al., 2019; Ndukwu and Onyeoziri, 2020).

A clearer visual understanding of how oil yield varies across the different drying methods is provided in Fig. 1. It

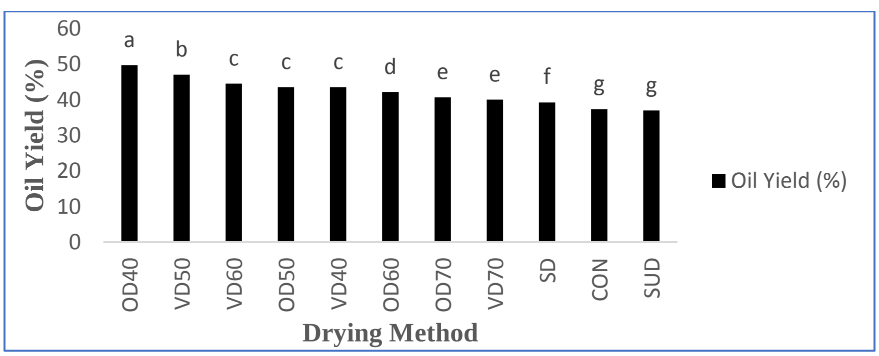


Figure 1: Bio-oil yield across drying treatments

highlights the strong performance of controlled drying techniques compared to natural drying methods. The large significant difference between oven drying at 40°C and sun drying is an indication that the controlled drying methods which have stable and regulated drying environments were more effective in preparing the seed matrix for oil extraction. This observation is consistent with findings by Chimsook and Assawarachan (2017), who also reported higher oil yields under controlled hot-air drying of avocado, compared to open-air drying due to better regulation of drying conditions.

The Fig. 1 also shows a gradual decline in oil yield with increasing temperature in oven drying, reinforcing the idea that excessive heat negatively affects oil stability. This visual representation complements the numerical data and aids quick performance identification trends among treatments.

3.2 Interaction Effect of Drying Method and Temperature

The combined effect of drying method and temperature for oven and vacuum drying is presented in Fig. 2, indicating a clear interaction effect. In oven drying, oil yield steadily decreased as temperature increased. This suggests that intense exposure to heat may have damaged oil components (Guillaume et al., 2018). This trend is however contrary to what was observed by Omosuli et al. (2017), where oil yield rose with first few increments in drying temperature, before declining with higher drying temperatures.

In contrast, vacuum drying showed an increased oil yield up to 50°C, followed by a decline at higher temperatures. This pattern suggests that moderate temperatures under reduced pressure likely created optimal conditions for oil extraction.

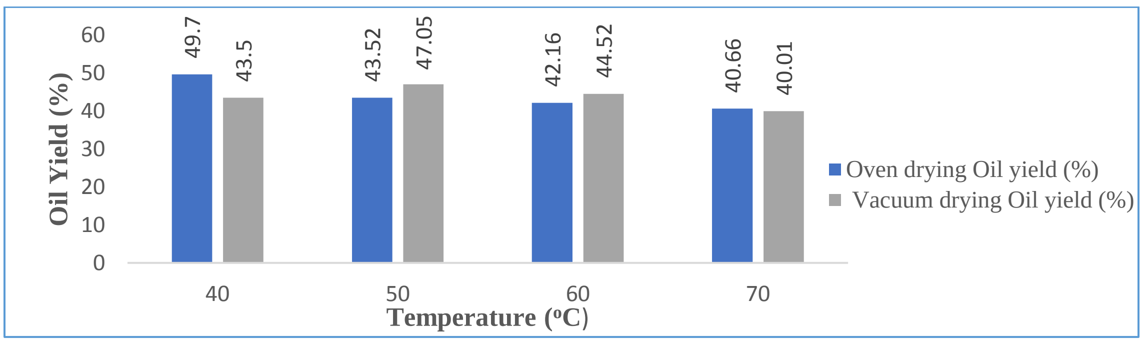
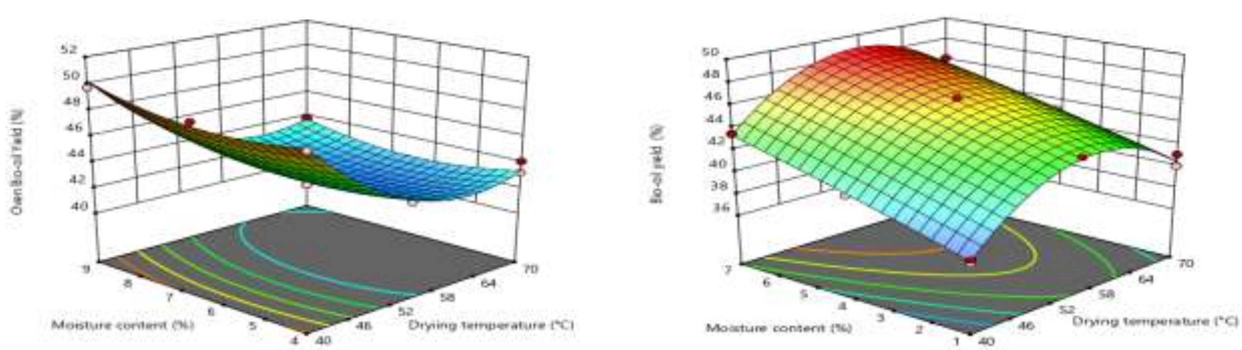


Fig. 2: Interaction effect of drying method and temperature on MSO yield`*

The different trends observed between the two methods confirm that drying method and temperature work together rather than independently. This aligns with improved quality and bioactive compounds which depended on the interaction between processing variables of different drying techniques (Belwal et al., 2022)

Moreso, Figs. (3a and 3b) present the chart showing the effects of controlled drying temperature and moisture on the bio-oil yield of molinillo seed. Fig. 3a shows that the highest oil yield of 49.7% was recorded at temperature of 40°C and moisture content of approximately 9% w.b, which, gradually decreased to the lowest oil yield of 40.66%, as the temperature increased to 70°C at a consequential lowest seed moisture content of approximately 4%. This suggests that at 40°C drying, it was possibly easier to remove moisture and partially alter the seed tissues' structure, which



(a) (b)

Fig. 3 Effect of (a) oven (b) vacuum drying temperature and moisture content of molinillo seed on the bio-oil yield

improved solvent access during extraction. The decrease in the oil yield as the drying temperature rose over this threshold, indicated the beginning of unfavourable thermal impacts, such as deterioration or loss of extractable oil components as was similarly observed by Zhang et al. (2018), hence, exhibiting a convex quadratic curve. The model showed that drying temperature was significant at both linear and quadratic levels, while, moisture content showed no significant effect with the range studied. However, the optimum oil yield of 50.22% was obtained at 40°C drying temperature and moisture content of 9.0% (w.b). The expression for oven drying which connecting the oil yield with the seed moisture content and drying temperature is shown in eq. (8):

$$\%\ \text{MSO yield} = 42.66 - 3.05t + 0.3908w - 0.1909tw + 2.94t^2 + 0.9998w^2 (adj.R^2 = 0.947) \quad (8)$$

In Fig.3b, vacuum drying however, showed a different trend by exhibiting a concave quadratic trend. This model showed that the drying temperature and the moisture content had linear significant effects, with the drying temperature also possessing quadratic significant effect. Here, the oil yield of 37.45% was least at the lowest drying temperature and moisture content of 40°C and approximately 1% respectively. Then, the oil yield gradually increased to its highest level of 48.34% at a drying temperature of 55°C and 7% moisture content. Thereafter, the yield began to drop with increase in drying temperature and lower moisture contents, likely due to excessive drying, which is similar to the trend as reported by Ndukwu and Onyeoziri (2020). Also, at temperatures below 55°C, drying of the oilseed seemed incomplete, thus, leaving residual moisture within the seed matrix, with limited solvent penetration and reduced extraction efficiency, as has been also, established by Zhou (2019). Optimum oil yield of 48.46% was obtained at drying temperature of 59.8% and approximately 6.8% (w.b) moisture content. The vacuum drying expression linking oil yield with those of seed moisture content and drying temperature is presented in eq. (9):

$$\%\ \text{MSO yield} = 46.19 + 1.25t + 2.71w - 0.1550tw - 4.29t^2 - 0.1641w^2 (adj.R^2\ 0.944) \quad (9)$$

3.3 Physicochemical properties of bio-oil

The result of the physicochemical properties of the bio-oil extract from the different drying methods and control are shown in Figs. (4 – 9). The drying technique used before extraction has a significant impact on the acid value of Molinillo (*Hura crepitans*) seed oil, underscoring the significance of appropriate seed stabilization. Fig. 4 shows that acid value which reflects an exact measure of triglyceride hydrolysis and the amount of free fatty acids released during processing, varied between 10.16 and 11.53 mg KOH/g, with the vacuum drying at 70˚C having the lowest value (10.16 mg KOH/g), whereas, sun drying and the control had the highest values of 11.19 mg KOH/g and 11.53 mg KOH/g respectively. The highest level of acidity exhibited by the control sample, signified the degree at which the completely withheld seed moisture promoted enzyme activity and accelerated oil decomposition. Sun drying exhibited

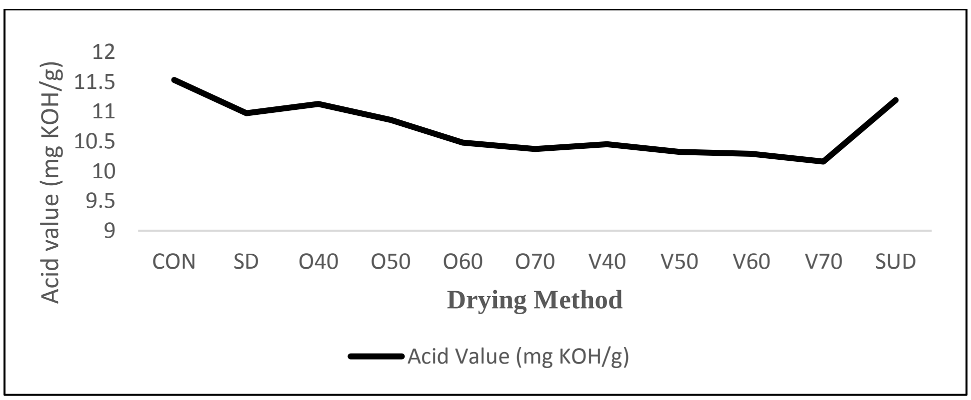


Fig. 4. The effect of drying method on the acid value of molinillo seed oil

a reduction in acidity, as was similarly demonstrated by Omosuli et al. (2017), but was slightly higher than that of the solar drying possibly due to its lesser exposure to heat and higher retained moisture. Moreso, there was a gradual decrease in acid value, as the oven drying intensity increased, hence, indicating more effective moisture removal and deactivation of enzymes. Trend of this nature has also been reported in the literature (Ndukwu and Onyeoziri, 2020; Mo and Li, 2021). Vacuum drying consistently produced the lowest acid values, even at higher temperatures. It was noticed that these variations in acid values from the drying processes were statistically significant. However, acid value from each of the extracted oils was higher than the value (4.41 ± 0.20 mg KOH/g) gotten from molinillo seed reported by Adewuyi et al. (2014); but slightly above the highest allowable limit of 10mgKOH/g for virgin oil (Omosuli et al., 2017). Vacuum drying, and to a lesser extent oven drying offered distinct benefit, nevertheless, these extracted oils could still be used for various industrial and non-edible uses, such as biodiesel production after free fatty acids reduction through pretreatment

Saponification is the level of oxidation involved at storage, which showcases oil deterioration. A rise in saponification value raises the oil volatility. Thus, improves the oil value as lower molecular weight of the present fatty acids in the oil is exhibited in a singular gram weight of oil, which could improve energy generation when ignited (Aremu et al., 2015). The saponification values of oil from the different drying treatments range from 193.55 to 200.3 mg KOH /g, as presented in the Fig. 5, which are comparable to that of jatropha (200 mg KOH /g), but higher than those of gmelina and chali seed oil with 181.31 and 191.40 ± 3.11 mg KOH/g respectively. Although drying method to some extent affected saponification value, but the values still fell within the range reported for most vegetable and non-edible seed oils (Ghada et al., 2015; Umeuzuegbu et al, 2020; Keneni et al., 2020). It can be observed that only the saponification value of the control fell within the recommended CODEX standard (185 - 195 mg KOH/g), but, the high saponification valued oils from the different drying method, especially those of oven and vacuum drying, however, could be very fit for liquid soap and shampoo manufacturing (Odoom and Edusie, 2015).

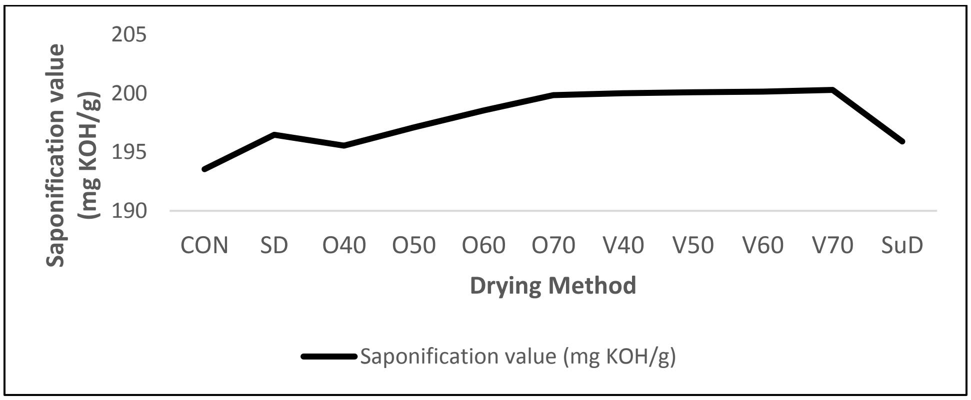


Fig. 5. The effect of drying treatments on the saponification value of molinillo seed oil.

Iodine value quantifies the level of unsaturation in an oil or fat. A decline in iodine value signifies dismantling of the double bonds which normally occur by oxidation or polymerization, particularly when heat is added. Non-drying oils posses' iodine value below 100 gI2/100g of oil. More extensively, an appropriate drying oil should have iodine value not less than 130 gI2/100g (Aremu et al., 2015).

The iodine values in this study which range from 83 to 87.5 as presented in Fig. 6, fell within the range of non-drying oil. They are comparably higher than those of gmelina (64.12 gI2/100g of oil) (Umeuzuegbu et al, 2020), African oil bean seed oil (16.81 gI2/100g of oil) (Ndukwu and Onyeoziri, 2020) and almond seed oil (2.65 gI2/100g of oil) (Aremu et al., 2015). This implies that they would not be useful in paint and ink formation but, could suitably be utilized for other industrial purposes such as soap production (Mishra, 2023).

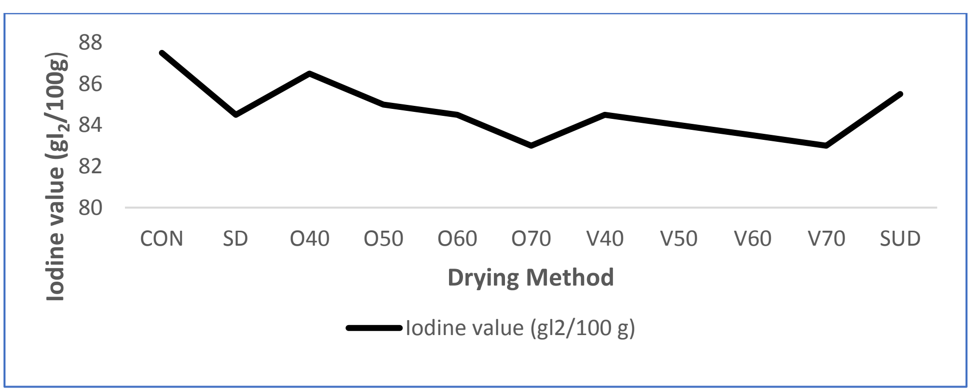


Fig. 6. Effect of drying method on the iodine value of molinillo seed oil

Peroxide value (PV) primarily portrays the level of lipid oxidation and as such, it is generally utilized in estimating stability of oil by referring to smaller values to have larger oxidative stability. This study showcases PV ranging from 1.839 to 1.888 meq/kg as shown in Fig. 7. It can be observed that drying method had no much effect on the PV of this oil. Nevertheless, the values are higher than that of melon seed oil (0.45meq/kg), but lower than those of African oil bean seed oil (3.8 – 5.2 meq/kg), gmelina seed oil (9 meq/kg) and much lower than that of Bambara groundnut (290 meq/kg) (Aremu et al., 2015; Umeuzuegbu et al., 2020; Ndukwu and Onyeoziri, 2020). In relation to the CODEX standard which allows for highest permissible limit of 10meq. of oxygen/kg, the range of values from this study, show very low rancidity. This suggests that, they may not be vulnerable to deterioration during storage and could suitably be utilized for industrial purposes such as biodiesel production.

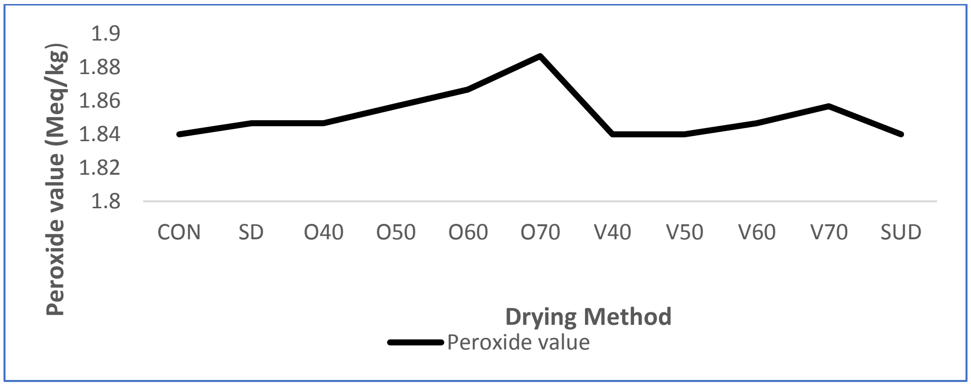


Fig. 7 Effect of drying method on the peroxide value of molinillo seed oil

Specific gravity obtained in this study ranged from 0.9190 to 0.9275 as can be seen in the Fig. 8, with very small differences observed, which, still fell within the range of common vegetable oils. The highest specific gravity of 0.9275 was exhibited by the control sample, followed by sun drying (0.9252), while the least was vacuum drying at 70˚C (0.916). The minimal variability among group suggests that drying conditions resulted in slight but detectable changes in oil density, most likely due to variations in retained moisture content along with small compositional changes rather than structural changes to the oil itself. Omosuli et al. (2017) has also reported similar trend (0.9032–

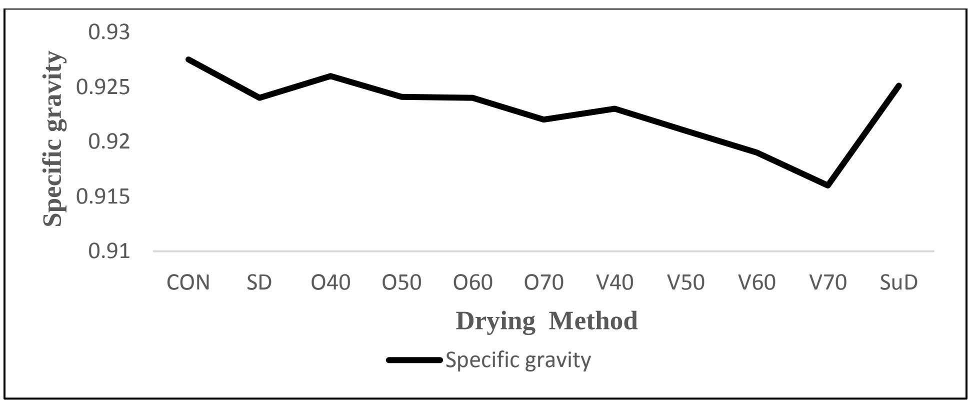


Fig. 8 Effect of drying method on specific gravity of molinillo seed oil

0.9075) in specific gravity of seed oil which also slightly decreased as a result of increasing intensity of drying temperature. However, the results were above the ASTM standard range. This indicated either the presence of oxygenated compounds or high molecular weight compounds which may not permit the oils to be expressly applied as bio-fuel in combustion engines, to avoid engine deposits which hamper atomization of the oil (Umeuzuegbu, 2020). As such, the oils can easily be applied to other non-fuel purposes, or receive some pretreatments before converting to biodiesel.

Flash point is the smallest temperature upon which a fuel releases adequate vapour that ignites with surrounding air at any slightest spark. Flash point of fuel is a very significant parameter that potentially guard against unexpected fire hazard during fuel manufacturing, movement, and even at storage (Fu, 2018). The results of flash point from the study are between 125 and 125.67˚C (Fig. 9), which are lower than the ASTM standard (≥130˚C). The flash point of the oven dried samples increased gradually with temperature increase, above those of the vacuum samples, which also, followed similar trend, whereas, the control and the sundried produced almost equal results. The values are also actually low compared to those of gmelina (Umeuzuegbu, 2020) and Jatropha (Ghada, 2015). However, the studied oils can suitably be handled and applied in cold regions; unlike these other high flash point oils which may not be able to suitably utilized in similar cool temperature environment (Okwundu et al., 2019).

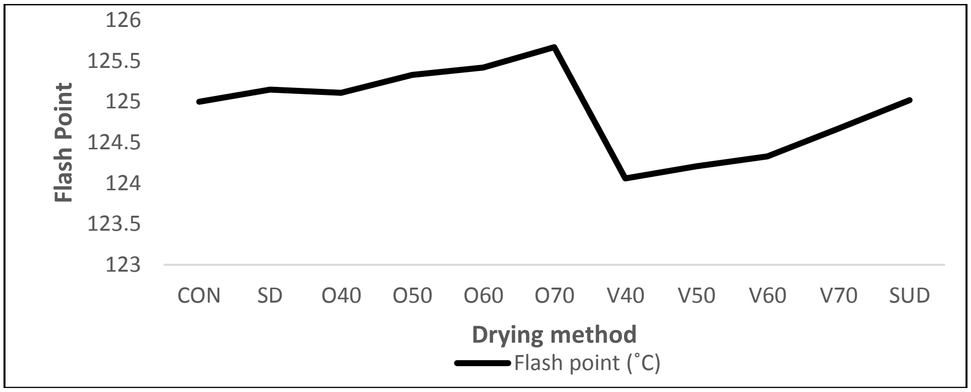


Fig. 9. Effect of drying method on flash point of molinillo seed oil

4.0 Conclusion

This study demonstrated that the process of drying molinillo seeds prior to oil extraction had a significant impact on the quantity and quality of oil extracted. For elimination of moisture more efficiently and shielding the oil from deterioration, controlled drying techniques, particularly vacuum drying, consistently performed better than solar and sun drying. Oil yield was highest with oven drying at 40°C. However, the oil yield was more stable with vacuum drying especially at 50°C and 60°C, and the quality criteria like acid value and peroxide value were also reasonably low at these temperatures, which thereby produced the best results. Raising the temperature too much decreased yield, perhaps, due to oil loss and thermal degradation.

According to the physicochemical findings, molinillo seed oil was found to be resilient and appropriate for a number of industrial uses, such as the manufacture of soap, biodiesel, and other non-edible products. Although several attributes marginally exceeded ASTM limitations for direct fuel use, basic precondition processes could easily remedy this. In general, the results portrayed molinillo seed as a potential but underdeveloped oil resource. Moreso vacuum drying at a moderate temperature provided a workable technique to optimize oil yield and quality, especially for the development of sustainable bioenergy in tropical locations like Nigeria.

5.0 Recommendation

The controlled drying methods which include vacuum and oven drying at mild temperatures (40–60 °C) are recommended, since they effectively remove moisture while preserving acceptable oil characteristics and enhancing extraction efficiency.

Future research should also concentrate on assessing the oil's fuel performance and storage stability.

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