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Physicochemical Properties of Oil Extracted from Selected Underutilized Oil Seeds

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Abstract:

Overdependence on the conventional sources of oil has led to a shortage of edible oils for both domestic and industrial uses. This has called for search for newer sources of oil with better quality to boost industrial activities. The aim of this work is to determine the physicochemical properties of oil extracted from the selected underutilized oilseeds. Oil from cassava seed, African star apple, alamanda seed, tiger nut, and water melon seed was extracted with petroleum ether. Physicochemical properties of oil were determined, proximate compositions of the oil seeds were determined using the AOAC methods while the fatty acid profile of the oilseeds were determined using gas chromatography. Cassava seed has the highest value for protein ($23.28 \pm 0.08\%$) and ash content (8.40 ± 0.02). Alamanda seed is a rich source of oil having a proximate composition of ($43.30 \pm 0.03\%$). Saponification value of the oilseeds for cassava seed, African star apple, alamanda seed, tiger nut, and water melon are $112.63 \pm 2.87 \text{MgKOH/g}$, $196.13 \pm 4.01 \text{MgKOH/g}$, $281.17 \pm 3.55 \text{MgKOH/g}$, $127.93 \pm 2.00 \text{MgKOH/g}$ and $250.33 \pm 2.02 \text{MgKOH/g}$ respectively. Iodine value for the oil seed is $>100 \text{g}$ except in cassava seed having a value of $87.24 \pm 2.80 \text{g/100g}$. The peroxide value of the oil seeds falls within the recommended value having a value less than 10Meq/g . The fatty acid profile of the oilseeds shows that they contain oil that is rich in polyunsaturated fats like ricinoleic and linoleic acid as well as monounsaturated fats such as oleic acid and saturated fats such as stearic acid and palmitic acid. The underutilized seed in this study could be a cheap source of oil for domestic and industrial uses for the production of soaps, detergents, cosmetics, animal feed production and lubricants for machines.

Keywords: Polyunsaturated, monounsaturated, physicochemical, edible, oil

1. Introduction

The industrialization of oilseeds is one of the most important agro-industrial activities in the world today. Oil from plant and animal are employed in the formulation of foods, cosmetics, and drugs in many industrial activities (Nimete *et al.*, 2011). These oils act as insulators to the body protective layer or internal organs such as heart and lung as well as energy source to the body in absence of carbohydrate. Vegetable oil is also used as a viable feedstock for biodiesel production (Ochigbo and Paiko, 2011). There is a serious shortage of edible oils and fats in the developing countries of the world, especially in Africa. This shortfall is being largely met by increased importation of edible oils from the developed countries of the world which is putting a heavy strain on the foreign exchange position of these African countries. This situation, therefore, calls for concerted research effort to increase edible oil production in Africa. In order to overcome this challenge, new sources of oil-bearing crops are being exploited (Sam *et al.*, 2008).

Seeds of plants are a good source of food for animals, including humans, because they contain nutrients necessary for plant's initial growth, including many healthy fats, such as omega fats which are in turn healthy for human consumption. In fact, the majority of foods consumed by human beings are seed-based foods. Edible seeds include cereals, legumes, and nuts. Oilseeds are often pressed to produce rich oils – sunflower, flaxseed, rapeseed, sesame.

Plant proteins play significant roles in human nutrition, particularly in developing countries where average protein intake is less than that required. Plant protein products are gaining increased interest as ingredients in food systems throughout many parts of the world and the final success of utilizing plant proteins as additives depends greatly upon the flavor characteristics that they impart to foods. Since oilseeds are valuable sources of lipids as well as proteins, numerous studies on protein functionality of major and minor oilseeds such as soybean (Molina *et al.*, 2004), peanut (Yu *et al.*, 2007),

rapeseed (Yoshieset *al.*, 2008) sunflower (Gonzalez and Vereijken, 2007), almond (Sze-Tao, 2000), winged bean (Zolgenet *al.*, 2005), have been reported.

2. Materials and Method

Oil seeds from alamanda seed, African star apple, Tiger nut, Cassava seed, and Water melon seed) were obtained from fruit market in Minna. The were dried for a period of 3weeks at room temperature and weighed to ensure that the weight is constant, i.e. the moisture content of the seeds were reduced signifying the seed are properly dried. After which they were dehusked manually. The seeds were then dried at room temperature and ground using mortar and pestle into powdery form. The powdered seeds were then preserved in the laboratory at room temperature.

2.1. Proximate Composition Analysis

The proximate analysis of the oil seeds for moisture, total ash, crude fiber, fat, protein, and carbohydrate were carried out in triplicate using methods described by (AOAC 2005).

2.2. Characterisation of Oil Obtained from the Different Oil Seeds

2.2.1. Specific Gravity

The specific gravity of the oil was determined by taking a known volume of the oil and weighing it on a weighing balance. The same volume of water was taken and weighed. The ratio of the weight of the oil of the same volume of water was calculated to obtain the specific gravity of the oil (Alhassan and Issa, 2000).

2.2.2. Density

The density of the oil was calculated using the pycnometer with the formula;

$$\text{Density} = \frac{M1 - M0}{V1}$$

Where M0 is the mass in gram of the pycnometer or density bottle, M1 at a temperature t1 and V1=volume in ml of the oil in the pycnometer (Egwim and Ibrahim, 2012).

2.2.3. Viscosity Test

The same amount of oil was drawn into the stem of the Cannon Fenske viscometer to the mark above the upper bulb of the viscometer. The time taken for the meniscus to fall between the upper and lower mark of the bulb through the capillary tube of the viscometer was recorded and used to determine viscosity.

2.2.4. Refractive Index

The refractive index was determined using an Abbe refractometer.

2.2.5. Determination of Acid Value

25 ml of diethyl ether and 25 ml of ethanol was mixed in a 250 ml beaker. The resulting mixture was added to 10 g of oil in a 25 ml conical flask and a few drops of phenolphthalein were added to the mixture. The mixture was titrated with 0.1 M NaOH to the endpoint with consistent shaking after which a dark pink colour was observed and the volume of 0.1 M NaOH (V_o) was noted. The acid value was determined using the formula;

$$\text{Titre (ml)} = \frac{\text{Acid Value} \times 4.0}{0.1 \times \text{weight of sample}}$$

Free Fatty Acid = Acid Value $\times 0.503$

(Laboratory Handbook, 1997)

2.2.6. Determination of Saponification Value

Two gram of the sample was weighed into a conical flask; 25 ml of 0.1 Nethanoic potassium hydroxide was then added. The content which was constantly stirred was allowed to boil gently for 60 min. A reflux condenser was placed on the flask containing the mixture and a few drops of phenolphthalein indicator was added to the warm solution and then titrated with 0.5 M HCl to the endpoint until the pink colour of the indicator disappeared. The same procedure was used for other samples and a blank. The expression for saponification value (SV) is given by:

$$SV = \frac{56.1 N (V_o - V_i)}{m}$$

where: V_o – the volume of the solution used for the blank test, V_i – the volume of the solution used for determination, N – actual normality of HCl used, m – mass of the sample.

2.2.7. Determination of Peroxide Value

To 1 g of the oil sample, 1 g of potassium iodide and 20 ml of solvent mixture (glacial acetic acid/chloroform, 2/1 by volume) were added and the mixture was boiled for one minute. The hot solution was poured into a flask containing 20 ml of 5% potassium iodide. A few drops of starch solution were added to the mixture and the latter was titrated with 0.025 N sodium thiosulphate and the peroxide value was determined as follows:

$$PV = \frac{S N 10^3}{W}$$

where: S – ml of $\text{Na}_2\text{S}_2\text{O}_3$, N – normality of $\text{Na}_2\text{S}_2\text{O}_3$,
 W – weight of oil sample (g).

2.2.8. Determination of Iodine Value

Into a conical flask, 0.5g of the sample was added and 20 ml of chloroform was added to dissolve the oil. Then 25ml of Iodine reagent was added to the flask using a safety pipette influenced chamber. A stopper was then inserted and the content of the flask was vigorously swirled. The flask was then placed in the dark for 30 min. At the end of this period, 20 ml of 10% aqueous potassium iodide and 100 ml of water were added using a measuring cylinder. The content was titrated with 0.1 M sodium-thiosulphate solution until the yellow colour almost disappeared. A few drops of 1% starch indicator were added and the titration continued by adding thiosulphate drop-wise until blue coloration disappeared after vigorous shaking. The same procedure was used for the blank test and for other samples. The iodine value (IV) is given was calculated with the formula:

$$IV = 12.69c(V1 - V2)m$$

where: c – concentration of sodium thiosulphate used,
 $V1$ – volume of sodium thiosulphate used for the blank,
 $V2$ – volume of sodium thiosulphate used for determination,
 m – mass of the sample.

2.3. GC Analysis

The fatty acid profile of the oil seeds was determined by gas chromatography (Hewlett Packard, model 5750).

3. Results and Discussion

3.1. Proximate Composition of the Oil Seeds

	Moisture (%)	Protein(%)	Lipid (%)	Fibre(%)	Ash(%)	Carbohydrate(%)
Cassava seed	7.17±0.63	23.28±0.88	29.85±0.02	2.81±0.03	8.40±0.02	27.83±1.87
African Star Apple	7.81±0.01	18.48±1.32	10.94±0.04	1.99±0.01	1.35±0.05	57.04±2.99
Alamanda Seed	6.21±0.03	17.09±1.19	43.30±0.03	2.27±0.10	2.50±0.25	27.97±1.85
Tiger Nut	8.21±0.01	11.9±0.02	29.74±0.64	6.02±0.03	1.60±0.20	42.53±0.80
water Melon	3.61±0.04	18.04±0.02	28.33±0.15	3.80±0.08	3.80±0.20	42.42±0.18

Table 1

The data above is the Mean±Standard mean of error of three replicates

Moisture content recorded for the seeds ranged from 3.61-8.21. All the oil seeds have good percentage of moisture content. The moisture content of the oil seed indicates that the seeds have a long shelf life when it is preserved. It is a good attribute of storage for the oilseeds. Crude protein of the oil seeds was highest in cassava seed (23.28±0.88), African star apple (18.48±1.32) and watermelon (18.04±0.02). The protein content of cassava seed was similar to that obtained by (Popoola and Yangomodou, 2006) where the crude protein show 20.20%. The high protein content recorded for cassava seed, watermelon and African star apple indicates that they are a rich source of protein. Lipid composition of the oil seeds for cassava seed, African star apple, alamanda seed, tiger nut and watermelon seed are 29.85±0.02, 10.94±0.04, 43.30±0.03, 29.74±0.64 and 28.33±0.15 respectively. The results showed that the underutilized oilseed in this study contains a high amount of oil which can be explored for domestic and industrial uses. Only African star apple contains a considerably less oil content to that of other oilseeds implying that more of the African star apple seed will be needed for oil extraction. Crude fibre represents that portion of food not used up by the body but mainly made up of cellulose together with a little lignin and is known to increase bulk stool (Eleazuet *et al.*, 2011). The fibre content obtained from the five oilseeds are: 2.81±0.03, 1.99±0.01, 2.27±0.10, 6.02±0.03, and 3.80±0.08, for cassava seed, African star apple seed, alamanda seed, tiger nut, and watermelon respectively. From the value obtained tiger nut was the highest in fibre content with a value of 6.02±0.03%, followed by watermelon having a value of 3.80±0.08. The result obtained from this work was similar to that obtained by (Gambo and Da'u, 2013). The high fibre content of tiger nut shows that the seed has a high carbohydrate content that cannot be metabolized maybe due to unavailability of digestive enzymes for such carbohydrate.

The ash content of the oil seeds for cassava seed, African star apple, alamanda seed, tiger nut, and watermelon are: 8.40±0.02, 1.35±0.05, 2.50±0.25, 1.60±0.20 and 3.80±0.08 respectively. The cassava seed has the highest ash content 8.40±0.02%, followed by 3.80±0.20% for water melon seed. Ash content is an indication of the mineral content of a food. This is an indication that the cassava and water melon seed have more mineral content than the other oil seeds studied here.

Carbohydrate content of the oil seed is showing a value range of 57.04-27.83 with African star apple having the highest carbohydrate value 57.04 ± 2.99 and cassava seed with the lowest value 27.83 ± 1.87 . Carbohydrate content of the cassava seed is $27.83 \pm 1.87\%$ and that of alamanda seed is $27.97 \pm 1.85\%$ which might be due to the high amount of lipid content which accounts for more of the percentage composition of the proximate composition of the alamanda seed. The high carbohydrate content found in African star apple, tiger nut, and watermelon, indicate that the oilseeds are a rich source of carbohydrate.

3.2. Physicochemical Properties of Oil Seeds

	Cassava Seed	African Star	Alamanda seed	Tiger Nut	Watermelon
% Oil yield	21.56 ± 2.99	17.50 ± 2.43	44.51 ± 4.14	28.64 ± 1.69	24.20 ± 0.69
Specific Gravity	0.67 ± 0.03	0.78 ± 0.02	0.89 ± 0.01	0.72 ± 0.00	0.79 ± 0.10
Density(g/cm ³)	0.66 ± 0.12	0.78 ± 0.11	0.77 ± 0.40	0.61 ± 0.06	0.74 ± 0.06
Acid Value(MgNaOH/g)	5.53 ± 0.64	4.00 ± 0.30	7.97 ± 0.40	7.87 ± 0.32	8.93 ± 0.64
FFA(MgNaOH/g)	2.78 ± 0.33	2.01 ± 0.15	4.00 ± 0.21	3.95 ± 0.16	4.49 ± 0.33
Saponification value(MgKOH/g)	112.63 ± 2.87	196.13 ± 4.01	281.17 ± 3.55	127.93 ± 2.00	250.33 ± 2.02
Peroxide Value(Meq/g)	9.17 ± 1.04	4.50 ± 0.50	7.82 ± 0.45	3.78 ± 0.34	11.97 ± 0.10
Iodine Value(g/100g)	94.74 ± 0.25	117.99 ± 0.40	125.44 ± 0.83	162.47 ± 2.21	151.16 ± 2.05
Viscosity at 33.5°C(Mpa)	16.00	25.00	22.00	24.00	26.00
Refractive Index	1.4560	1.4500	1.4560	1.4600	1.4520

Table 2

The value obtained is mean $\pm$ Standard error of mean of triplicate

3.2.1. Physicochemical Properties of Oil

Refractive index of an oil is the ratio of the speed of light at a defined wavelength to its speed in the oil/fat itself. This value varies with wavelength and temperature, the degree of unsaturation, the type of substitutions of component fatty acids and with accompanying substances. Refractive index is commonly used in quality control to check for the purity of material and to follow hydrogenation and isomerization (Hoffman, 1986). The oil extracted from five different oil seed samples namely; cassava seed, African star apple, alamanda seed, tiger nut, and watermelon, all have their refractive index within the range of 1.4500-1.4600 which is within the acceptable range of 1.4677 to 1.4707 according to Codex Standard for fats and oils from plant source (CODEX-STAN, 1999). The refractive index of the oil samples indicates the level of optical clarity of the oil sample relative to water and also that the oil is of good purity.

The specific gravity of the five oilseed samples have shown to have specific gravity which is less dense than water as such, they will be very essential in cream production as it will make the oils flow and spread easily on the skin (Oyelekeet *et al.*, 2012). According to Yahaya *et al.* (2012), specific gravity is basically used in conjunction with other parameters in assessing the purity of oil. Specific gravity obtained in this work, indicate that the oil may find application in cream production.

The density of seed oils is dependent on their fatty acid composition, minor components, and temperature (Fakhir and Qadir, 2011). The density of the oil seeds are 0.67 ± 0.03 , 0.78 ± 0.02 , 0.89 ± 0.01 , 0.72 ± 0.00 , 0.79 ± 0.10 respectively for cassava seed, African star apple, alamanda seed, tiger nut, and watermelon. The density of the oil is less than that of water this implies the oil will float in water. Viscosity of oils seeds increases with the molecular weight and decreases with increasing unsaturated level and high temperature (Nourrechnet *et al.*, 1992). The viscosities of the oil seeds were determined at room temperature (33.50°C). The viscosity of the oil seeds show a relatively low viscosity; 16mpa.s, 25mpa.s, 22mpa.s, 24mpa.s, and 26mpa.s for cassava seed, African star apple, alamanda seed, tiger nut, and watermelon respectively. The low viscosities indicate that the oil obtained from the oil seeds are light and may be highly unsaturated. (Nangbeset *et al.*, 2013).

Acid value is an indication of the quality of fatty acids in oil. Low acid value in oil indicates the oil will be stable over a long period of time and protect against rancidity and peroxidation. (Oyelekeet *et al.*, (2012); Popoola and Yangomodu, (2006); Gambo and Da'u (2014). The acid values obtained are 5.53 ± 0.64 , 4.00 ± 0.30 , 7.97 ± 0.40 , 7.87 ± 0.32 , 8.93 ± 0.64 (MgNaOH/g) for cassava seed, African star apple, alamanda seed, tiger nut, and watermelon respectively. The values are compared favourably with Codex recommended values for virgin olive oil (Kirk and Sawyer, 1991). The relatively low acid value indicates limited lipase hydrolysis of the oils, would give some expectations of low value for diacylglycerols and monoglycerols. Acid values depict the edibility of an oil and suitability for use in the paint and soap industries (Aremuet *et al.*, 2006). The Acid values obtained from this work is an indication that the oils can serve both domestic purpose and may also find application in the production of paints, liquid soaps and shampoos (Akintayo (1997), Aremuet *et al.*, (2000).

Saponification value is a measure of oxidation during storage. An increase in saponification value in oil increases the volatility of the oils. It improves the quality of the oil because it shows the presence of lower molecular weight components in 1g of the oil which will produce more energy on combustion (Engler and Johnson, 1983). Saponification value obtained from this work indicated that the oils contain high proportion of lower fatty acids due to the high saponification value obtained., 112.63 ± 2.87 MgKOH/, (cassava seed), 196.13 ± 4.01 MgKOH/g (African star apple), $281.17 \pm$

3.55MgKOH/g (Alamanda seed), 127.93±2.00MgKOH/g(tiger nut), 250.33± 2.02MgKOH/g(water melon). The oils are having high saponification value thereby contain lower proportion of fatty acids and can be regarded as edible oils and may find application in the production of soaps and shampoo.

Iodine value is a measure of the degree of unsaturation. It is an important characteristic of seed oils, which determines its applicability in soaps, cosmetics industries (Hamilton, 1999). It is also used to quantify the number of double bonds present in the oil which reflects the susceptibility of the oil to oxidation. The iodine value of the five oil seeds shows a value greater than 100gI₂/100g except in cassava seed with an iodine value of 94.74g/100g. The oil can be regarded as liquid oil and find its application in the manufacture of soap (Kochhar, 1998). According to Anhwangeet *et al.*, (2010), oil with iodine value that ranges between 100 and 150 has good properties of absorbing oxygen on exposure to the atmosphere. This makes the oil useful for soap manufacturing due to its thick, sticky consistency which does not form a dry film. The high iodine value also indicates that the oil seed has drying property, having more of unsaturated fatty acids than saturated fatty acids and can find its application in vegetable oil based ice cream production.

Peroxide value is an indicator of lipid oxidation. High value of peroxide value is an indicator of high level of rancidity of the oils and also suggests absence or low levels of antioxidants. WHO/FAO (1994) stipulated a peroxide level of not more than 10mequivalent of oxygen/g of the oils, for it to be edible. The peroxide value of the five oil seeds fall within this range with an exception of that of water melon with 11.97± 0.10Meq/g, this is an indication that the oil obtained from water melon seeds may have a high level of oxidative rancidity with a low level of antioxidants. High quality oils are low in free fatty acids (Overhult *et al.*; 1994). An excessive amount of free fatty acid lowers the smoke point of oil and will cause "popping" of the oil during smoking. The free fatty acids of the five oil seeds show a value less than 5.00MgNaOH/g.

3.3. Fatty Acid Profile of the Oilseeds

Samples	Linoleic acid µg/g	Arachidonic acid µg/g	Oleic acid µg/g	Recinoleic acid µg/g	Palmitoleic acid µg/g	Stearic acid µg/g	Linolenic acid µg/g	Total fatty acids
Cassava seed	10.909	2.169	12.960	25.201	0.315	10.622	9.193	71.369
African star	8.182	4.770	12.625	31.562	0.315	7.986	8.090	73.529
Alamanda seed	12.273	1.302	5.196	12.989	0.287	16.438	9.921	58.404
Tiger nut	16.273	8.671	14.081	32.399	0.655	18.467	8.268	98.813
Water melon	9.545	3.903	12.591	31.478	0.481	13.258	8.050	79.307

Table 3

The fatty acid profile shows that the oil contains polyunsaturated fats (linoleic, arachidonic, recinoleic and linolenic), monounsaturated fats (oleic acid) and monounsaturated fats (palmitoleic and stearic acid). It contains polyunsaturated fatty acid in the percentage of 66.5% for cassava seed, 82.7% for African star apple, 62.4% for alamanda seed, 66.4% for tiger nut and 66.8% for watermelon seed. Recinoleic acid is a polyunsaturated omega -9- fatty acid having a hydroxyl functional group on the 12th carbon atom. This functional group causes recinoleic acid to be more polar than most fats (Beitz, 2005). It is a fatty acid that is valuable in feedstocks (Wilson, 1998).

Linoleic acid is a polyunsaturated omega -6- fatty acid. It is a carboxylic acid with an 18-carbon chain and two cis bonds, with the first bond located at the sixth carbon from the methyl end (Anneken *et al.*, 2005). It belongs to one of the two families of essential fatty acids, which means that the human body cannot synthesize it from other food components, it is therefore pertinent to get it from dietary sources. (Burr *et al.*, 1930). In the industry, this oil can be used in making quick drying oils, which are useful in oil paints and varnishes. These applications exploit the easy reaction of the linoleic acid with oxygen in the air, which leads to cross-linking and the formation of a stable film called linoloxyn. It has also become increasingly of use in cream production because of its inflammatory properties on the skin (Darmstadt *et al.*, 2002). Linolenic acid is another essential fatty acid which is necessary for human health as they cannot be produced within the human body. The values recorded for linolenic acid for the oilseed were very close. Linolenic acid is very vital for human health. The oil seeds can be seen as a good source of linolenic acid which cannot be synthesized from stearic acid due to the absence of 12 and 15 desaturase enzyme.

Oleic acid was also recorded to be present in all the oilseeds studied. The values recorded are as follows in the highest order; tiger nut (14.081µg/g), cassava seed (12.960µg/g), African star apple (12.625µg/g), watermelon seed (12.59µg/g) and alamanda seed (5.196µg/g). Oleic acid is a monounsaturated fat it is found to be part of human diet, it can find application also in soap production as an emulsifying agent, also as an emollient (Carrasco, 2009). It is also used as an excipient in pharmaceuticals and as a solubilizing agent in aerosol products (Smolinske, 1992). It therefore suggests that the oil from these underutilized oilseeds could find application in several industries.

The oilseeds in the present study also contain stearic acid. The stearic acid recorded for tiger nut was the highest 18.467µg/g, alamanda seed 16.438µg/g, watermelon seed 13.158µg/g, cassava seed 10.622µg/g, and African star apple 7.986µg/g. Stearic acid was the highest fatty acid profile found in alamanda seed with a considerably high amount in tiger

nut and watermelon seed. Stearic acid is a saturated fatty acid with an 18 carbon chain. Its major application can be in the production of detergents, soaps, and cosmetics such as shampoos and shaving creams.

The oil extracted from the oilseeds was relatively low in palmitic acid. Palmitic acid is a saturated fatty acid with a 16 carbon atom, its consumption as part of human diet increases low density lipoprotein. Palmitic acid can undergo hydrolysis to produce glycerol and sodium palmitate which are useful in soap production. Low level of this fatty acid can help to prevent accumulation of low density lipoprotein when the oil is consumed.

4. Conclusion

From the study of the five oil seeds namely; Cassava seed, African star apple seed, alamanda seed, tiger nut, and watermelon, it can be observed from the physicochemical properties of the oil, that the oil extracted from the various oils seeds can serve as a good substitute to conventional oils. The oil is high in nutritional properties and can be used as edible oils but may need further purification to make it good for human health. The industrial uses of the oil are enormous as it can find application in soap, shampoo, paints, varnishes, cosmetics, lubricating oil as well as source of oil for biodiesel production. The oil harvested from the oilseeds is rich in polyunsaturated fats which are healthy fats for human consumption as they provide the body with low density lipoprotein. The oilseeds will not only be of benefit to the industry and human consumption, they will also serve economic purposes for the farmer who will mass produce them to meet with local and export demand. The findings of this study work will also be of great help to the economy and environmental protection as seeds which are seen as waste products will no longer be found littering the streets but will serve as a source of income and employment.

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