



Material and compressional properties of *Irvingia gabonensis* (O'Rorke) Bail polymers.

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ABSTRACT

The physicochemical, material and compressional properties of the *Irvingia gabonensis* (O'Rorke) Bail polymer were evaluated and compared with xanthan gum and hydroxypropylmethylcellulose (HPMC). The *Irvingia* polymer was extracted using established methods and processed using two methods of drying, that is, oven-drying and freeze-drying. The compression mechanisms were evaluated using the Heckel and Kawakita equations. The mechanical strength of the tablets was evaluated using crushing strength and friability. The results showed that the *Irvingia* kernel polymer was slightly acidic, free of heavy metals, with irregularly shaped particles that exhibited some degree of crystallinity. The *Irvingia* kernel polymer was directly compressible and formed intact non-disintegrating tablets similar to the standard polymers. The Heckel and Kawakita equations indicated that the *Irvingia* polymer deformed plastically with fast onset and a high amount of plastic deformation compared to xanthan gum and HPMC. The freeze-dried *Irvingia* gum showed significantly ($p < 0.05$) higher crushing strength and lower friability than the oven-dried polymer. The results indicated that the drying method had a significant impact on the compression characteristics as well as the properties of the polymer tablets. Thus, the *Irvingia* polymer could be more suitable for the formulation of tablets using direct compression.

KEY WORDS: Polymer, *Irvingia gabonensis*, tablets, Compression properties, Heckel equation, Kawakita equation

INTRODUCTION

Polymers are used as excipients in pharmaceutical formulations to regulate the release of drugs from in dosage forms. Several natural polymers have been characterized and evaluated as useful pharmaceutical excipients in various drug delivery systems. Notable among them are alginates, carrageenan, xanthan gum,

guar gum, karaya gum, albizia gum, khaya gum and terminalia gum, which have been found useful as disintegrants, binders, emulsifiers and matrix systems (1-5). Plant polymers are hydrophilic and can hydrate quickly in cold water and achieve high viscosity and low concentration. They have demonstrated comparable activity to official ones, and in some cases, even superior characteristics. Due to their natural origin, easy availability, biodegradability, and lack of toxicity, they are appealing and suitable alternative pharmaceutical excipients to the synthetic polymer.

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Understanding the drying process is important in foods and pharmaceuticals since it can cause undesirable changes in the density, porosity, texture, sorption characteristics, and overall performance of the dry product (6). The type and condition of the drying process have been shown to influence the properties of the final product since if a large amount of moisture is removed materials have been shown to behave differently depending on the drying process (7). The most commonly used drying techniques for plant gums include oven drying (8), spray drying (9), freeze-drying (10), and vacuum drying (11).

Irvingia gabonensis (O'Rorke) Bail (family *Irvingiaceae*) also known as African bush mango or wild mango, *Ogbono* (Igbo), *Aapon* (Yoruba), *Mbukpabuyo* (Efik), *Ogni* (Ibibio) and *Okpukpa* or *Egbene* (Izons), belongs to a genus of African and South-East Asian rees (12, 13). *Irvingia gabonensis* bears edible fruits that resemble mango and is valued for their fat and protein-rich nuts. The nuts also known as dika nuts are used widely in Nigeria for thickening soups because they form a thick viscous sauce base when heated.

Different parts of the *Irvingia* plant have been utilized in herbal medicine for curing a variety of ailments including hernias, yellow fever, dysentery and poisons (14). The antimicrobial, analgesic, anti-diarrheagenic and anti-ulcer properties of the plant parts have been demonstrated (12, 14, 15). The kernel or nuts and bark extract have been observed to reduce fasting blood glucose levels in both test animals and humans thus its possible usefulness in the management of diabetes, bodyweight reduction and blood cholesterol level control (16).

Irvingia seed contains lipids and polymeric substances and studies have shown that the lipids could be useful as a suppository base (17), tableting lubricant (18) and sustained release agent (19) while the mucilage has been used as emulsifying and suspending agents (20), polymer for microbead formulations (21) and tablet binder (22). When used as a binding agent in metronidazole tablets, the tablets possessed lower

mechanical strength and slower drug release properties than gelatin standard binder (22). Even though the physicochemical properties of the *Irvingia* seed oil have been evaluated, the physicochemical, materials and compression properties of the mucilage have not been fully characterised to determine their potential usefulness as a directly compressible tablet excipient. In addition, the effects of the drying method on the properties have not been evaluated. The physicochemical, material and compressional properties of *Irvingia* mucilage have been evaluated in this study in comparison to xanthan gum and hydroxypropylmethylcellulose (HPMC).

MATERIALS AND METHODS

Materials

The materials used were xanthan gum (Myprotein Co, Chesire, UK), hydroxypropylmethylcellulose (Ranbaxy Laboratories Ltd, Gurgaon, India) and *Irvingia* mucilage derived from the *Irvingia gabonensis* kernel, also known as the dika nut and sourced from the local market at Okolobiri - Gbarain, Yenagoa, Bayelsa State Nigeria.

Extraction of the *Irvingia* polymer

The dried kernels of *Irvingia gabonensis* (without the seed coats) were ground using a laboratory mill and macerated in petroleum ether for 24 hours. The fat was separated from the residue and the residue was macerated in petroleum ether repeatedly until no more fat was obtained. The final residue (deemed to be fat-free) was soaked in double strength chloroform water BP for 5 days before being precipitated with ethanol. Diethyl ether was used to wash the precipitated gum after it had been filtered and the purified gums were dried for 48 hours in a hot air oven (Laboratory oven TT-9083, Techmel and Techmel, TX, USA) at 40°C. The dried polymer was pulverized and stored in airtight containers (22). The freeze-dried polymer was prepared by freeze-drying the purified polymer in a freeze dryer (Christ GmbH, Osterode, Germany) at -84°C and a pressure of -0.371 bar for 24 hours.

Phytochemical and physicochemical properties

Phytochemical constituents

Using standard procedures, the following phytochemical tests were performed on *Irvingia* kernel polymer: mucilage, phenolics, alkaloids, chloride, sulphate, and carbohydrates (23, 24).

Elemental constituents

The determination of the elemental constituents of *Irvingia* kernel polymer was carried out using an atomic absorption spectrophotometer (AAS, model 2500 Torontech, Inc., Toronto, ON, Canada) (25).

Proximate composition

The crude fat, crude fibre, ash and starch contents were determined according to the methods of the Association of Official Analysis Chemists (26). The crude protein content was estimated from the nitrogen content determined by elemental analysis using a conversion factor of 6.25 (27).

The Ph of the *Irvingia* kernel polymer

A pH meter (pH tutor, Eutech instruments, Rajah Crescent, Singapore) was used to determine the pH of a 1% aqueous dispersion of *Irvingia* kernel polymer.

Fourier transform infrared spectroscopy

The FTIR spectrum of the *Irvingia* kernel polymer was recorded using a Jasco FTIR spectrophotometer (Model 4100, Jasco corporation, Ishikawa-Co, Hachioji, Tokyo, Japan). For the spectroscopy, the dry polymer was blended and pressed into a pellet using the potassium bromide (KBr) technique and analysed by scanning the sample at a wavelength range of 400 to 4000 cm^{-1} (28).

X-ray powder diffraction

A Shimadzu X-ray diffractometer was used to obtain the diffraction pattern for the *Irvingia* kernel polymer (Model XRD 6000, Kyoto, Japan). This was carried out

at room temperature using copper made material at 40 kV and 30 mA voltages and current, respectively, and a diffraction angle range of 4° to 90° with a scanning speed of 10 deg/min (21).

Scanning electron microscopy

Scanning electron microscopy (SEM) was carried out using a JSM 6390 (JOEL, Tokyo, Japan) scanning electron microscope at 15 kV voltage. The process was performed by placing the powdered polymer on an aluminium stub and then coated with gold using a JFC ion sputter (JFC – 1100, JOEL, Tokyo, Japan).

Particle size determination

Particle size distribution of the polymer gum was carried out using a laser light diffractometer with a dry feeder (Sympatec Rodos SR 544, Sympatec, Germany) and the mean particle size was determined.

Moisture Content determination

The moisture content (%) of the polymers was determined using TGA (Pyrus 1 Thermogravimetric Analyzer, TGA 7, Perkin Elmer Instruments, U.S.A.). The samples were placed in ceramic pans and heated at a rate of $10^\circ\text{C}/\text{min}$, from 60 to 300°C . They were held for 1 minute at 60°C .

Density measurements

The particle density (ρ_t) of all polymers was measured using the liquid pycnometer technique using xylene as the displacement liquid (1). The polymer's loose bulk density (ρ_o) was determined using established methods (1, 29). The tapped density was measured by tapping 20 g polymer placed in a 50 ml measuring cylinder 100 times at a standard rate of 38 taps per minute (BS 1460). The relative density at zero pressure, D_o , was obtained from the ratio of the loose bulk density and particle density. All determinations were made in triplicate.

Compressibility and flowability characteristics

The compressibility of the polymers was assessed

using Carr's index and Hausner ratio (30). The angle of repose to measure the polymer flowability was determined using the fixed funnel method.

Preparation of tablets

Direct compression was used to prepare *Irvingia* polymer tablets ($500 \text{ mg} \pm 10 \text{ mg}$) from both oven-dried and freeze-dried polymers while xanthan gum and hydroxypropylmethylcellulose (HPMC) were used as standards. A carver hydraulic hand press (Model C, Carver Inc., Menomonee falls Wisconsin, USA) was used to compress the weighed polymer powder for 30 seconds at predefined loads in a 10.5 mm die with flat-faced punches. The die and punches were lubricated with magnesium stearate in acetone (1% w/v) before compressing each tablet. Upon ejection, the tablets were stored in sealed containers for 24 hours over silica gel to enable elastic recovery and hardening. Their weights (within 1.00 mg) and dimensions (0.01 mm) were determined using a Mettler electronic balance and a micrometre screw gauge, respectively. The relative density, D , of the tablet was calculated using Equation 1:

$$D = \frac{\frac{w}{V_t}}{\rho_t} \quad \text{Eq. 1}$$

where, w = tablet weight (g), V_t = tablet volume, and ρ_t = particle density (g/cm^3).

Compression properties

The polymers' compression properties were analyzed using the Heckel and Kawakita equations (1, 31). The Heckel equation is utilized to calculate the relationship between the powder bed relative density during compression, D , and the applied pressure, P , shown in Equation 2:

$$\ln \left[\frac{1}{(1-D)} \right] = KP + 1 \quad \text{Eq. 2}$$

where, K is the slope of the linear portion, which is

inversely related to the material's mean yield pressure, P_y . The intercept, A , was used to compute the relative density, D_A , using Equation 3 (32):

$$D_A = 1 - e^{-A} \quad \text{Eq. 3}$$

The relative density D_B , which describes the phase of rearrangement at low pressures is obtained from Equation 4:

$$D_B = D_A - D_O \quad \text{Eq. 4}$$

Powder compression is studied using the Kawakita linear model (33). The degree of volume reduction (C) was calculated using Equation 5:

$$C = \frac{(V_o)}{V_o} = \frac{abP}{(1+bP)} \quad \text{Eq. 5}$$

where, V_o is the initial bulk volume and V_p is the bulk volume after compression. Equation 5 can be rewritten as Equation 6:

$$\frac{P}{C} = \frac{P}{a} + \frac{1}{ab} \quad \text{Eq. 6}$$

where, the constant a defines the minimum porosity of the material before compression, whereas b represents its plasticity. The pressure term P_k is obtained by taking the reciprocal of b (1).

Tablet properties

DBK tablet hardness tester (Model EHO1, DBK instruments, Mumbai, India) was used to determine the tablet crushing strength. The tablet was positioned between the spindle and anvil, then the knob was turned to apply a diametric force to the tablet, and the force (in Newton) needed to divide the tablet in half was recorded. The result is given as the mean ($n = 6$) and standard deviation.

Tablet friability was determined using Cambell electronic Thermonik friability apparatus (Model C-FTA 20, Mumbai, India). The tablets (10) were

weighed and placed in the friabilator and the friabilator was rotated for 4 minutes at 25 RPM. The tablets were reweighed, and the percentage loss in weight (friability) was recorded. The analysis was carried out in triplicate.

RESULTS AND DISCUSSION

Phytochemical and physicochemical properties

The percentage yield of *Irvingia gabonensis* kernel polymer was 43.8 %^w/_w, which is quite appreciable for a natural product. The results of the phytochemical analysis of *Irvingia gabonensis* kernel polymer, presented in Table 1, showed that the *Irvingia* polymer contains carbohydrates, reducing sugar, mucilage and alkaloid but no chloride, sulphate and phenolic compounds. The presence of carbohydrates and reducing sugars in the *Irvingia gabonensis* kernel polymer indicates that the polymer is a polysaccharide.

The elemental constituents of the *Irvingia* polymer, given in Table 2, showed that the polymer does not contain heavy metals such as lead, copper and arsenic. This is a very desirable feature as their presence would have limited its use both pharmaceutically and in the food industry. The mineral composition is influenced by the soil type on which the plant is grown, thus there could be variation from samples of the same species planted on different soil types (34).

The proximate compositions of the *Irvingia gabonensis* kernel polymer, presented in Table 3, showed the presence of protein, fat, fibres and carbohydrates.

Table 1 Phytochemical screening of *Irvingia* kernel polymer

COMPONENTS	OVEN-DRIED IRVINGIA	FREEZE-DRIED IRVINGIA
Phenolic compound	-	-
Alkaloid (Mayer,Wagner&Dragendorff's)	+	+
Chloride (10% lead acetate solution)	-	-
Sulphate (barium chloride drops)	-	-
Reducing sugar (Fehling's solution)	+	+
Carbohydrate (Molisch's test)	+	+

Table 2 Elemental constituents of *Irvingia* kernel polymer

ELEMENT (mg/100 g)	OVEN-DRIED IRVINGIA	FREEZE-DRIED IRVINGIA
Calcium (Ca)	23.54	22.88
Iron (Fe)	1.15	1.19
Magnesium (Mg)	0.33	0.27
Potassium (K)	266.77	288.50
Sodium (Na)	34.54	33.30
Zinc (Zn)	1.23	1.20
Copper (Cu)	< LOD	< LOD
Manganese (Mn)	1.10	1.08
Lead (Pb)	< LOD	< LOD
Arsenic (As)	< LOD	< LOD
Phosphorous (P)	333.24	321.60
Selenium (Se)	< LOD	< LOD
Cadmium (Cd)	< LOD	< LOD

LOD: Limit of detection

There was no significant difference in the proximate composition of the oven- and freeze-dried *Irvingia* polymer. The proximate compositions indicate that the polymer exhibited relatively high levels of protein, fat and starch content indicating that the composition is multicomponent. The fat content could be the deeply embedded fat in the polymer matrix which could not be extracted using the maceration process. The presence of fat could be an added advantage since the fats may also serve as a lubricant during compression which would eliminate the need for a lubricant since *Irvingia* fat has been shown to possess the ability to lubricate the die during tablet production (18).

The pH of the *Irvingia gabonensis* kernel polymer at a temperature of 30.5°C was 5.23 for the oven-dried sample and 5.80 for the freeze-dried polymer. *Irvingia gabonensis* kernel polymer is thus acidic. The drying process does not significantly affect the pH of the polymer. The pH of an excipient is important because the stability and physiological activity of most preparation are determined by their pH (35).

The Fourier transform infrared (FTIR) spectrum of *Irvingia gabonensis* polymer shown in Figure 1 showed several functional groups. It is possible to divide the

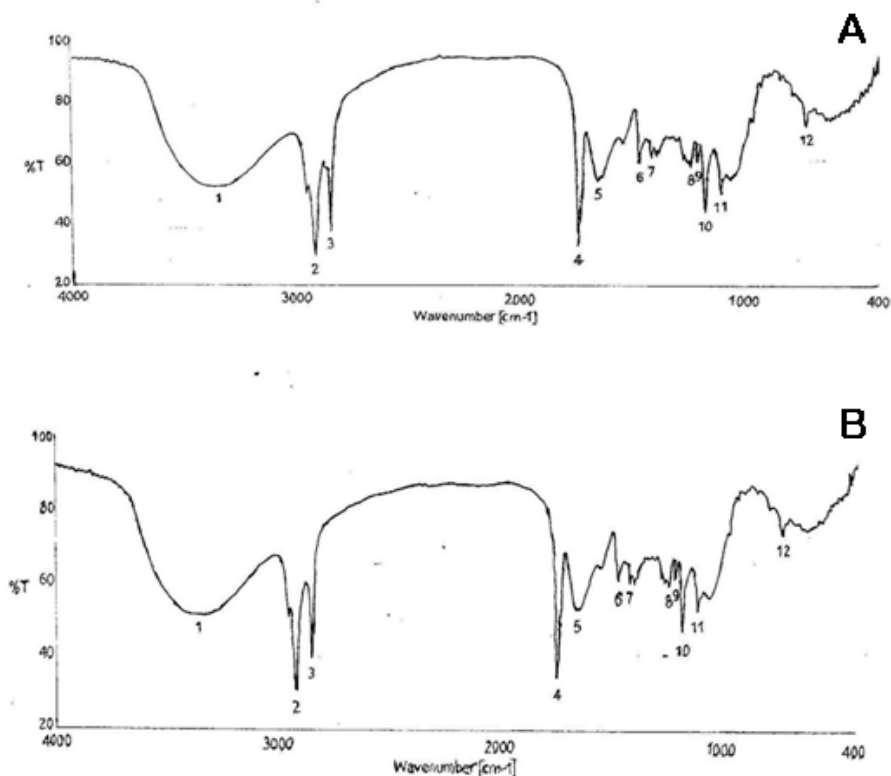


Figure 1 FTIR Spectrum of oven-dried (A) and freeze-dried (B) *Irvingia gabonensis* kernel polymers.

spectrum into four regions, the first, second, third and fourth regions, which range from 4000 to 2500, 2500 to 2000, 2000 to 1500 and 1500 to 400, respectively. The first, second and third regions are the functional group region while the fourth region is the fingerprint region. The fingerprint region contains a large number of

Table 3 Proximate composition of *Irvingia* kernel polymer

COMPONENT	COMPOSITION (%)	
	Oven-dried <i>Irvingia</i>	Freeze-dried <i>Irvingia</i>
Protein	12.88	12.72
Fat	2.77	2.68
Ash	2.85	2.86
Fibre	1.34	1.35
Starch	2.39	2.68
Reducing sugar	6.65	6.95

absorption peaks that accounts for a variety of single bonds. If the peaks of an IR spectrum of a sample (both the fingerprint region and the other regions) are identical or superimposable to the IR spectrum of another sample, then the two samples or molecules are identical (36). The functional group region of the *Irvingia* kernel polymer showed five distinct absorption peaks: the broadband at 3351.68 to 3358.48 resulting from hydrogen bondings that contribute to the complex vibrational stretches arising from free inter and intra-molecular bound hydroxyl groups (O-H) that makes up the entire structure of carbohydrates. Hydrogen bonding has a strong influence on peak shape and intensity, generally causing peak broadening ranging between 3600 to 3200 cm^{-1} (37, 38). The sharp absorption peak at 2916.81 was due to C-H aliphatic stretch; 2850.27 for the C-H alkane stretch; 1741.41 for the carbonyl C = O stretch; 1654.62 (oven-dried) and 1651.73 (freeze-dried) for the C = O stretch of ketone group and/or C = C stretch of alkene (27, 35). The fingerprint region of the *Irvingia gabonensis*

kernel polymer FTIR spectrum peaks are at 1175.4 and 1105.01 are due to C-O stretching of carboxylic acid, ether, ester or alcohol, suggesting the presence of these moieties and the absorption at 720.28 due to the C-H bending of phenyl ring substitution indicating the presence of an aromatic group (28, 38).

The x-ray diffraction patterns of *Irvingia gabonensis* kernel polymer presented in Figure 2 showed that *Irvingia* polymer is slightly crystalline. Amorphous materials usually exhibit a broad diffraction pattern, whereas the crystalline sample displays sharp diffraction peaks (21). The amount and intensity of each type of scattering in the X-ray diffraction peaks suggest polymer crystallinity. The results indicate that *Irvingia gabonensis* polymer exhibit some degree of crystallinity as shown by the intensity of the peaks, with the freeze-dried polymer being slightly more crystalline than the oven-dried polymer. The crystallinity of polymers has been shown to directly influence the compressional properties and mechanical strength of materials.

Material properties

The scanning electron micrograph of *Irvingia* polymers shown in Figure 3 shows the morphology and surface characteristics of the polymers. The particles of *Irvingia gabonensis* kernel polymer were mostly irregular in shape and appear flaky. Powder compaction properties are affected by particle shape; the more irregularly shaped the particles of a material, the greater the proportion of particles fragmenting during compaction, and the greater the compactibility (3). Furthermore, particle irregularity allows for closer packing during compression, resulting in greater tablet strength.

The material properties of the polymers are shown in Table 4. Particle size has been shown to influence polymer dispersion and hydration as well as the packing properties. The ranking of the particle size was freeze-dried *Irvingia* > oven-dried *Irvingia* > HPMC > xanthan. *Irvingia* gum had a significantly ($p < 0.05$) higher particle size and wider particle size distribution (data

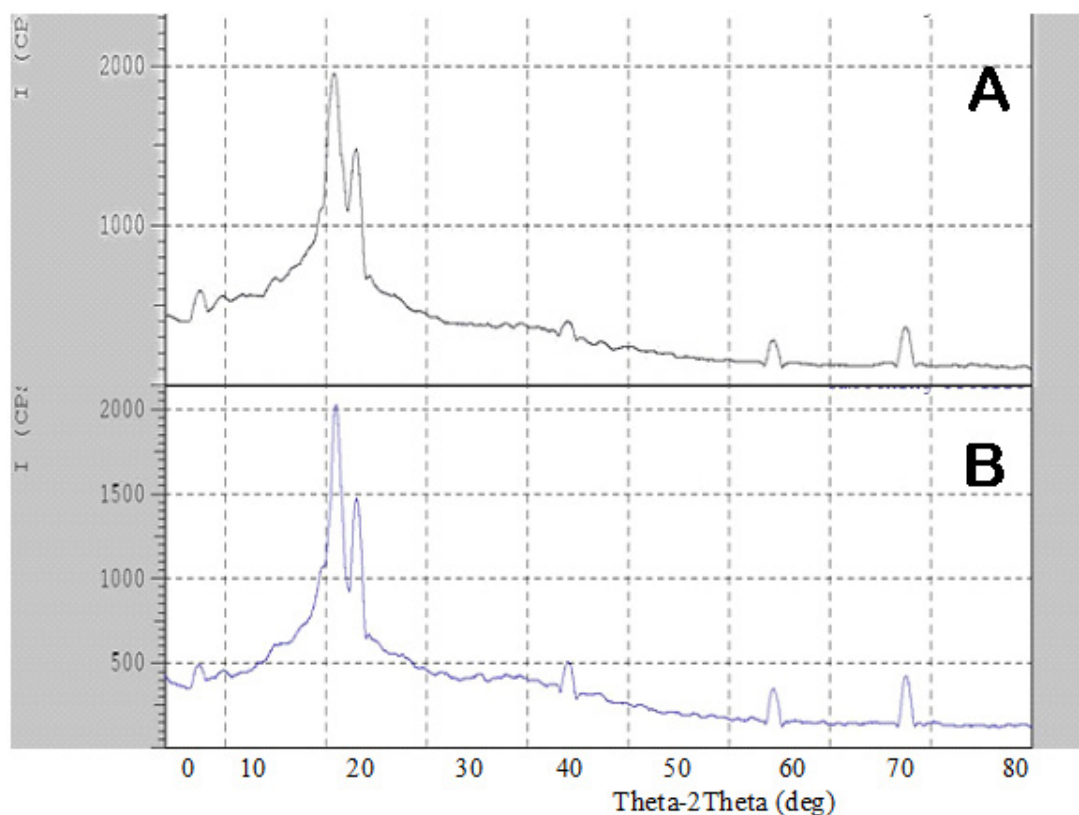


Figure 2 X-ray powder diffraction spectrum of oven-dried (A) and freeze-dried (B) *Irvingia* kernel polymers.

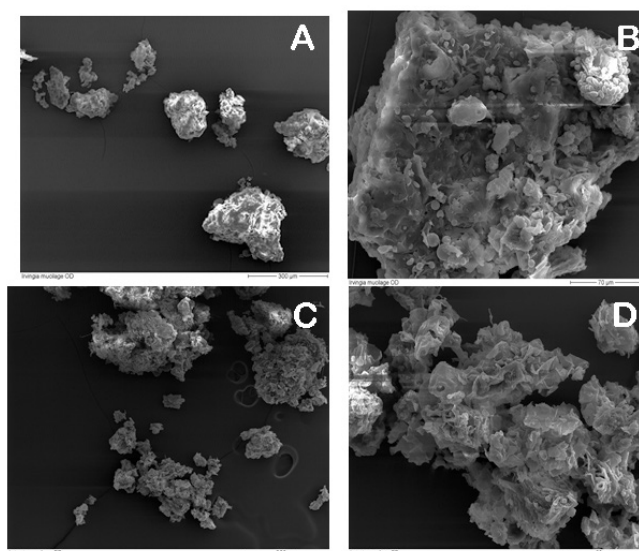


Figure 3 SEM of oven-dried (A and B) and freeze-dried (C and D) *Irvingia gabonensis* polymers at x 50 and x200 magnification, respectively.

not shown) than the HPMC and xanthan gum. It has been shown that as the particle size of polymer gums increases, the gums become more easily dispersible but hydrate more slowly (21).

Polymer gums are hygroscopic, suggesting they absorb moisture from the air based on their physicochemical properties and the humidity levels of the environment. Pharmacopeial limits for moisture content of natural gums such as acacia and tragacanth have been set at $\leq 15.0\%$ (39). Higher levels of water can lead to

microbial spoilage and subsequent deterioration in polymer quality. The moisture contents of the polymers were within the pharmacopoeial limits. However, *Irvingia* gums showed significantly ($p < 0.05$) higher moisture content than HPMC and xanthan gum. This indicates that *Irvingia* gum should be carefully stored to prevent the absorption of moisture.

Powder compaction behaviour is influenced by particle density, as dense, hard materials may require greater compression pressure to form cohesive and less friable tablets, whereas materials with low particle density would produce more cohesive compacts than those with higher values (1). The ranking of particle density was xanthan > HPMC > freeze-dried *Irvingia* > oven-dried *Irvingia*, indicating that xanthan gum would require greater compression pressure than HPMC and the *Irvingia* polymer to produce cohesive compact. The oven-dried *Irvingia* polymer also had lower particle density than the freeze-dried *Irvingia* polymer indicating that it would compact better at lower compression pressure.

The bulk and tapped densities, on the other hand, provide an understanding of the particle packing and rearrangement when zero or low pressure has been applied. It describes powder packing behaviour during tabletting unit operations such as mixing, granulation, die filling, and compression (1, 5). Higher bulk density is beneficial in tabletting since it minimises die fill volume. The rankings of bulk and tapped densities

Table 4 Material properties of *Irvingia* kernel polymer, xanthan gum and hydroxypropylmethylcellulose (HPMC)

PARAMETER	Oven-dried <i>Irvingia</i>	Freeze-dried <i>Irvingia</i>	Xanthan gum	HPMC
Particle size (μm)	138.5 ± 5.6	142.3 ± 6.8	75.5 ± 3.6	92.2 ± 2.7
Moisture content (%)	13.0 ± 3.4	12.0 ± 2.6	5.0 ± 0.4	6.0 ± 1.2
Particle density (g/cm^3)	1.203 ± 0.07	1.233 ± 0.03	1.509 ± 0.05	1.294 ± 0.04
Bulk density (g/cm^3)	0.306 ± 0.03	0.298 ± 0.02	0.388 ± 0.01	0.344 ± 0.03
Tap density (g/cm^3)	0.394 ± 0.02	0.382 ± 0.08	0.531 ± 0.06	0.440 ± 0.05
Hausner's ratio	1.29 ± 0.20	1.28 ± 0.14	1.37 ± 0.16	1.29 ± 1.04
Carr's index (%)	22.43 ± 0.14	21.88 ± 0.09	26.92 ± 0.05	22.46 ± 1.01
Angle of repose ($^\circ$)	54.83 ± 0.12	51.14 ± 0.07	55.12 ± 0.07	52.80 ± 0.07

were xanthan > HPMC > oven-dried *Irvingia* > freeze-dried *Irvingia*. The results indicated that *Irvingia* kernel polymers with larger particle sizes showed lower bulk and tapped densities while xanthan gum with the lowest values showed the highest values. There were significant ($p < 0.05$) differences in the density values of the polymers. The freeze-dried polymer had lower bulk and tapped densities than the oven-dried polymer. This is in agreement with other studies that showed that the powder packing and rearrangement were altered by the drying methods (40).

Hausner ratio measures interparticle friction, whereas Carr's index measures a powder's ability to decrease in volume, which is a measure of powder compressibility. Both the Hausner ratio and Carr's index are useful in predicting powder flowability. The lower a material's Carr index, the better its flowability but the worse its compressibility (32). Carr indices greater than 21, and Hausner ratios greater than 1.25 were reported for all the polymers, indicating poor flowability but better compressibility (30). The oven and freeze-dried *Irvingia* kernel polymers had closely comparable Hausner ratio and Carr's index to HPMC but values lower than for the xanthan gum (Table 4).

The angle of repose is a quantitative measure of the internal cohesion and coefficient of friction impact under relatively low loading, such as that experienced during powder blending, tablet dies or capsule shell filling (3). Angles of $\leq 30^\circ$ are typically indicative of free-flowing materials, whereas angles of $\geq 40^\circ$ indicate poor flow (41). The angle of repose of all the polymers was higher than 40° in the rank order

of xanthan > oven-dried > HPMC > freeze-dried *Irvingia* gum (Table 4). There were no statistically significant ($p > 0.05$) differences between the angle of repose of the different polymers. The results showed that the polymers are cohesive and not free-flowing and therefore would require a glidant when used as a directly compressible excipient to aid the flow of the powder from the hopper to the die.

Compression properties

The *Irvingia* kernel polymer was highly compressible and it formed intact tablets at all compression pressure used in this study like the standard polymers, xanthan gum and HPMC. The Heckel plots for the polymers are shown in Figure 4 while the parameter derived from the plots are presented in Table 5. The linearity of the plots ($r \geq 0.980$) indicates that the polymers deform mainly by plastic deformation. The ranking of the relative density at zero pressure (D_0) was HPMC > xanthan > oven-dried *Irvingia* > freeze-dried *Irvingia* polymers. The result indicates that HPMC exhibited the highest degree of packing in the die because of die filling while *Irvingia* polymer exhibited the lowest values. This indicates that *Irvingia* polymer is bulkier and would occupy more space in the die than the standard polymers. In addition, oven-dried *Irvingia* polymer exhibited higher bulk density compared to the freeze-dried form. This is similar to the results obtained for other gums (40, 42).

On the other hand, the rankings of D_A and D_B , which represents the total degree of packing at zero and low pressures, and the particle rearrangement phase in the

Table 5 Parameters derived from density measurement, Heckel and Kawakita plots and tablet properties

POLYMER	HECKEL PLOT				KAWAKITA PLOT		CRUSHING STRENGTH	FRIABILITY	CSFR
	DO	P_v (MPa)	D_A	D_B	P_k (MPa)	D_i	(N)	(%)	
Oven-dried <i>Irvingia</i>	0.254	333.33	0.817	0.563	1.754	0.398	55.70 $\pm$ 1.05	0.60 $\pm$ 0.02	92.83
Freeze-dried <i>Irvingia</i>	0.242	370.37	0.811	0.569	0.620	0.332	92.00 $\pm$ 0.95	0.38 $\pm$ 0.01	242.12
Xanthan gum	0.257	217.37	0.678	0.421	6.265	0.423	ND	0.01 $\pm$ 0.00	-
HPMC	0.266	400.00	0.779	0.513	0.969	0.290	ND	0.00	-

ND: Not determined – tablet remained intact at maximum crushing strength of 500N

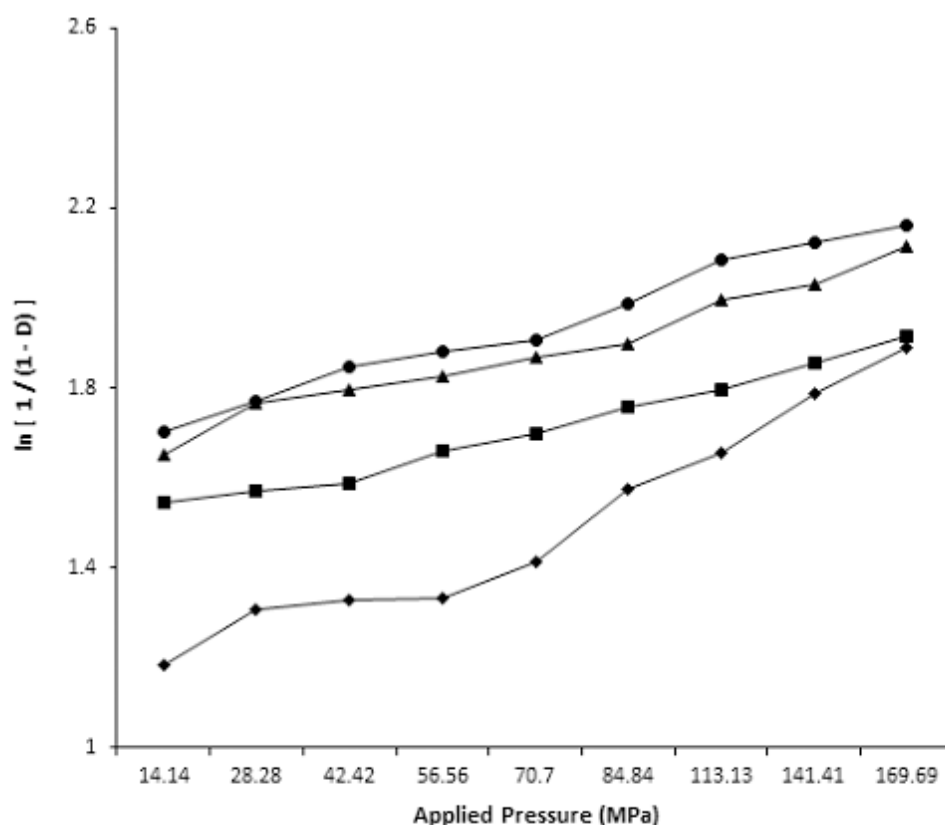


Figure 4 Heckel plots for the *Irvingia* kernel polymers ● oven-dried; ▲ freeze-dried; ◆ xanthan gum and ■ HPMC.

early compression, respectively, were oven-dried > freeze-dried > HPMC > xanthan. The result implies that the *Irvingia* polymer generally exhibited the lowest degree of initial packing in the die as a result of die filling but the highest rearrangement of the particles at the pre-compression stage and total degree of packing at zero and low pressures than the standard polymers (1). In addition, the *Irvingia* particles would undergo fragmentation to a greater degree while xanthan gum would exhibit the lowest degree of fragmentation, although fragmentation can occur simultaneously with plastic and elastic deformation of particles. These parameters were influenced by the method of drying i.e whether oven-dried or freeze-dried, with oven-dried polymer showing more fragmentation than the freeze-dried form.

The mean yield pressure P_y is inversely proportional to a material's ability to deform plastically under pressure. Materials that are brittle or easily fragmenting are

known to have high P_y (yield pressure) values while those that deform plastically or elastically exhibit typically low yield pressure (3). The ranking for P_y was HPMC > freeze-dried *Irvingia* > oven-dried *Irvingia* > xanthan gum. This suggests that xanthan gum had the quickest initiation of plastic deformation, while HPMC had the slowest. In addition, the P_y value for the oven-dried *Irvingia* polymer was lower than the freeze-dried suggesting that the oven-dried polymer would deform plastically more rapidly than the freeze-dried polymer. Therefore, on a high-speed tablet machine, the oven-dried polymer will deform plastically more readily under compression pressure and form intact tablets at a lower pressure than the freeze-dried polymer.

The Kawakita plots of the polymers shown in Figure 6 indicate a linear relationship with a correlation coefficient of 0.999 at all compression pressures. The slope and intercept of the plots give the constants a and b, respectively. The powder bed's minimum

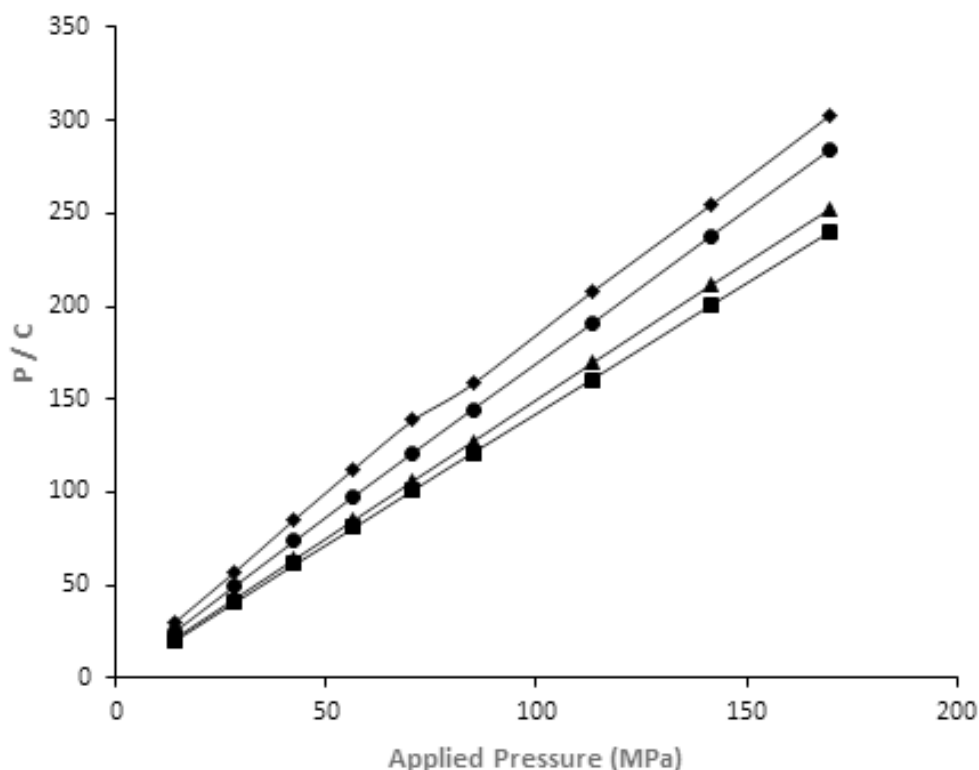


Figure 5 Kawakita plots for the *Irvingia* kernel polymers ● oven-dried; ▲ freeze-dried; ◆ xanthan gum and ■ HPMC.

porosity before compression, a , and the coefficient of compression, b , are associated with the plasticity of the materials. D_i , the initial relative density of the formulation is obtained by subtracting a from 1, while the reciprocal of b yields a pressure term, P_k , which describes the pressure needed to reduce the powder bed by 50% (3). The parameters obtained from the Kawakita plots are presented in Table 5.

The D_p , which is a quantification of the polymer's packed initial relative density when small pressures or tapping are applied, was in the rank order of xanthan > oven-dried > freeze-dried > HPMC. This indicates that the packed initial density of *Irvingia* gum is between the two standard polymers with the oven-dried polymer having higher values than the freeze-dried form. These values are also higher than the corresponding values of loose initial relative density, D_0 and are in line with previous findings (1).

The ranking of the P_k values, which are inverse measures of the quantity of plastic deformation that occurs during the compression process, was also xanthan > oven-dried *Irvingia* > HPMC > freeze-dried *Irvingia*. There were significant differences between the P_k values for all the polymers. It has been demonstrated that the lower the P_k value, the greater the total plastic deformation during compression (31). Thus, freeze-dried *Irvingia* polymer with the lowest P_k value demonstrated the most plastic deformation, while xanthan gum had the lowest values.

Tablet properties

The result of the tablet properties at relative densities of 0.85 which is a representation of commercial tablets shown in Table 5 indicates that the crushing strength of HPMC and xanthan could not be

determined because the tablets remained intact at the maximum crushing strength (500 N) while freeze-dried *Irvingia* polymer showed significantly ($p < 0.05$) higher crushing strength than the oven-dried polymer. The tablets also exhibited friability values of $< 1\%$ suggesting the tablets could withstand the rigours of abrasive motions during handling.

Crushing strength-friability ratio (CSFR) has been used to determine the mechanical strength of pharmaceutical tablets, the higher the CSFR, the stronger the tablet. The CSFR value of freeze-dried *Irvingia* tablets was significantly higher than those of oven-dried polymer. This indicates that the drying method had a significant impact on the compressional characteristics as well as the tablet properties of the polymer. Oven drying uses heat to evaporate moisture from the sample, leaving behind dry powder, whereas freeze-drying uses a vacuum to sublimate water (in the form of ice) under vacuum, which is useful for heat-sensitive materials (42). Thus, freeze-dried *Irvingia* polymer would be more suitable for the formulation of tablets by direct compression.

CONCLUSION

The results of this study showed that the physicochemical and material properties of the *Irvingia* kernel polymer were comparable to existing polymers. The *Irvingia* polymer was directly compressible and formed intact, non-disintegrating tablets similar to those made with existing polymers. The Heckel and Kawakita equations indicated that the *Irvingia* polymer deformed plastically with fast onset and a high amount of plastic deformation compared to xanthan gum and HPMC. Freeze-dried *Irvingia* polymer showed significantly stronger mechanical properties than the oven-dried polymer. The results indicated that the drying method had a significant impact on the compressional characteristics as well as the properties of the polymer tablets. *Irvingia* polymer could be suitable for the formulation of tablets by direct compression.

DECLARATION OF INTEREST

The authors declare no conflict of interest.

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