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Comparative Tableting Properties of Three Local Potato Starches II: The Mechanical Strength and Lamination Tendencies of Tablets

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ABSTRACT

The study was aimed at comparing the mechanical strength and lamination tendencies of three local potato (Sweet, Kaffir and Irish) starches. The compressional properties of the formulation were analysed using density measurement and assessed by Heckel and Kawakita equations while the lamination tendencies were assessed by the brittle fracture index (BFI). Tablets produced with potato starches have higher mechanical strength as seen in their tensile strengths. Sweet potato starch showed superiority over other potato starches in that respect. The lamination tendency was lower with corn starch BP than the potato starches. Starches obtained locally from potato tubers produced stronger tablets and hence can be used in formulation of tablets.

Key words: potato starches, lamination tendency, tensile strength, Heckel equation

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INTRODUCTION

The compaction properties of pharmaceutical powders are separated into two distinct terms, i.e. the compressibility as the ability of the powder to deform under pressure and the compactibility as the ability of a powder to form coherent compacts. There is growing research in the development of pharmaceutical excipients locally; therefore it is pertinent to study both compressibility and compactibility of new excipients.

Tensile strength and brittle fracture index (BFI) are two mechanical properties which have been used in assessing the usefulness of new binders in a formulation¹, tensile strength (T or T_o) is expressed mathematically as

$$T(\text{or } T_o) = 2F/\pi Dh \quad 1$$

Where F = force, D = diameter and h = height, T and T_o are tensile strength of normal tablet (without a hole) and T_o a tensile strength of tablet with a hole.

The BFI, is a measure of the lamination tendency of tablets² and calculated as

$$BFI = 0.5 (T/T_o - 1) \quad 2$$

The Heckel equation

The phenomena and mechanisms involved during compaction of pharmaceutical materials became an increasingly important concept in the design and development of solid dosage forms³. Many researchers have carried out studies on compaction characteristics of pharmaceutical powders with many equations and expressions reported⁴⁻⁷. However the Heckel equation is one of the most widely used equations for describing the compaction properties of powders⁸⁻¹². The Heckel equation analyses the ability of granules to undergo volume reduction, i.e. compressibility. It describes the relationship of the compact, density to the applied pressure¹¹. The Heckel equation which relates the relative density D of a powder bed during compression to the applied pressure P, provides information on the mechanism of powder consolidation during compact formation by the equation

$$\ln(1/1-D) = kP + A \quad 3$$

The Heckel plot is $\ln(1/1-D)$ plotted against applied pressure P in Newton per meter square (NM⁻²). It shows the mode of consolidation, fragmentation, plastic or elastic deformation or a mixture of any of the processes mentioned above.

The slope of the plot, k is the reciprocal of the mean yield pressure P_y which is the force required to attain plastic deformation. From the intercept A, the relative density can be calculated using

$$D_A = 1 - e^{-A} \quad 4$$

The relative density of powder at the point when the applied pressure is equal to zero (D_0) is used to describe the initial rearrangement phase of densification as a result of die filling with high value indicating very dense packing¹³. D_B describes the phase of rearrangement at low pressure, the extent of which depends on the theoretical point of densification at which particle deformation begins, and is expressed as

$$D_B = D_A - D_0 \quad 5$$

The limitation of Heckel equation as reported¹⁴ was that at low pressure it does not explain the compaction profile.

Kawakita and Ludde⁷ came with an equation used to study powder compression using the degree of volume reduction; C. the equation describes the relationship between the volume reduction of powder column and the applied pressure by the equation

$$C = [V_0 - V/V_0] = [abP/1 + bP] \quad 6$$

Where V_0 is the initial volume, V is the volume of powder column under the applied pressure P . 'a' and 'b' are constants characteristic to powder being compressed.

Equation 6 can be rearranged as

$$P/C = P/a + 1/ab \quad 7$$

When P/C is plotted against P , the constants can be evaluated as 'a' is given as reciprocal of the slope from the linear portion of the plot and equivalent to the value of C at very high pressures. $1/ab$ is the intercept. The actual meaning of the constants a and b are put to question¹⁵ however, Constant a , gives an indication of the maximum volume reduction available and is considered to describe the compressibility of a powder, while b is considered to describe an inclination toward volume reduction.

The limitation of Kawakita equation is that, the compaction process can be described only up to a certain pressure above which the equation is no longer linear¹⁶.

Many researchers^{17,18} treated their compaction data using both Kawakita and Heckel equations in order to compare the applicability of these methods. Hersey and Rees¹⁹ suggested that the Kawakita equation does not yield as much information as the Heckel equation. But others^{16,17} proposed the use of both methods together, in order to describe the mechanisms of compaction more accurately since linearity is observed at low pressure with Kawakita equation and at high pressures with the Heckel equation. Therefore both Heckel and Kawakita equations would be used in this study so that the two limitations can be taken care of.

The aim of this study is to compare the mechanical and compaction properties of three local potato starches with conventional maize starch BP.

MATERIALS AND METHODS

Paracetamol (May and Baker (Nigeria), Maize starch and Talc (B.D.H. Laboratories, U.K) and magnesium stearate Hopkin and Williams, U.K.). They were all utilized as obtained. Sweet, Irish and kaffir potatoes were purchased from local market. The three potatoes were identified by Prof. S. Sanusi, a taxonomist of Department of Biological Sciences, University of Maiduguri, Nigeria.

Extraction

Extraction and characterization was carried out as described earlier²⁰. Kaffir potatoes were thoroughly washed and all foreign materials were removed. The potato was peeled allowed to steep in water for about 24 hours; the steeped potato was pulverised using Philips blender (cucina HR1757, Japan). Enough quantity of water was added to the pulp which was then passed through an 180 μ m sieve. The filtrate was allowed to settle and 0.1N sodium hydroxide was added to separate the starch and proteinous materials as well as to neutralize the prevailing slight acidity. Excess sodium hydroxide was removed by washing several times with distilled water.

The clear supernatant fluid was poured away while sedimented starch was collected on a tray and air-dried on a table at room temperature. Using pestle and mortar the dried starch lumps were ground and fine powder passed through 180 μ m sieve.

Same was done for sweet and Irish potato, but the potatoes here were sliced with knife before they were pulverised.

Preparation of compacts

Paracetamol tablets (500 ± 10 mg) were prepared from the 250-1000 μ m size fraction of granules using varying binder concentrations (0-10% w/v) by compressing them at dwelling time of 30 sec with 9 predetermine loads on a Apex hydraulic press (Type 14, Apex Construction Limited, London) using 12.5mm die and flat faced punches lubricated with 2% w/v dispersion of magnesium stearate in ether-ethanol (1:1) prior to compression. Below 24.52 MNm⁻² crumbly tablets were formed and above the pressure 220.65 MNm⁻² the tablets were laminating. Hence, these pressures were considered the lower and upper limits.

Determination of tensile strength

To form tablets with center hole similar punches but with a center hole (upper punch) and a center pin (lower punch) were used²¹. Tablets with central hole were produced.

The tablets produced were kept- air tight container for 48 hours for further analysis to avoid false low yield¹⁸. The tensile strengths of tablet with or without hole were then calculated using equation 2.

Determination of Brittle Fracture Index (BFI)

The BFI was calculated for each formulation using equation 3. The determination of the tablets diameter and thickness were measured in triplicate using Digital Vanier calipers (Fisher Scientific, London).

Determination of Tablet Packing Fraction (P_f)

The packing fraction of the tablets from each size fraction was calculated from the particle densities of tablet compositions (i.e. Paracetamol and Maize starch) previously determined by xylene displacement method²⁰.

Results and Discussion

The mechanical properties of pharmaceutical tablets are important test for pharmaceutical tablets that often form part of manufacturer's own specification which is quantifiable by the tensile strength, Brittle fracture index, Heckel and Kawakita equations as well as crushing strength and friability.

The tensile strength of tablets increases with increase in either compression pressure or binder concentration. For brittle fracture to occur, the ratio of $T/T_o = 3$. By subtracting 1 and multiplying by 0.5, the maximum BFI value is 1 (Unity). The BFI value thus has a range of 0 (no. fracture tendency) to 1 (maximal fracture tendency). Tablets samples with BFI value (> 0.5) display a high fracture incidence while BFI values (< 0.5) has low fracture incidence during actual tableting¹⁰.

Tables 1 and 2 show the results of Tensile strength and BFI for the starches. The tensile strength of Paracetamol tablet formulations increased but the BFI slightly decreased with increase starch concentration. Irish potato starch exhibited highest BFI. The ranking is Irish $>$ sweet $>$ maize $>$ kaffir. When binder concentration was kept constant and compression pressure increased from 98.07 to 171.62 MNm⁻². The brittle BFI increased for all the starches the ranking was Corn $<$ Kaffir $<$ Sweet $<$ Irish potato starch. At compression pressure 98.07 MNm⁻² all the starches except Irish potato starch gave BFI of less than 0.5. That is low tendency to lamination and capping. A low BFI is desirable for minimum lamination and capping during tablet production, but the effect on tensile strength largely depends on the intended use of the tablets¹⁰. This could be explained, increase in binder concentration increases more bonds while increase in compression pressure results in further consolidation or additional strength of the bonds and hence more force is required to diametrically break the tablets. The tensile strength and applied pressure or binder concentrations are presented in figures 1 and 2 respectively. The tensile strength increases with both applied pressure and binder concentrations. Moreover, the values of T are inversely related

to the values of P_k of various formulations, and are probably responsibly for the tablets higher T values because greater total plastic deformation creates more contact points for inter-particulate bonding producing stronger tablets²². The decrease can also be explained by increased packing as a result of increase in binder concentration the ranking was, generally similar Corn < Irish < Kaffir < Sweet. Thus, Sweet potato starch formulation exhibited highest tensile strength as a result of both increased in binder concentration and increased in applied pressure.

Table 1: The result of tensile strength and Brittle Fracture Index of Paracetamol tablet formulation using different compression pressure

Starch	Applied pressure (MNm ⁻²)	T _o	T	BFI
Kaffir	98.07	0.416	0.792	0.452
	122.58	0.436	0.886	0.516
	147.10	0.450	0.978	0.587
	171.62	0.486	1.144	0.677
Sweet	98.07	0.452	0.875	0.468
	122.58	0.495	1.004	0.515
	147.10	0.539	1.186	0.600
	171.62	0.579	1.286	0.611
Irish	98.07	0.409	0.818	0.501
	122.58	0.423	0.965	0.641
	147.10	0.441	1.142	0.794
	171.62	0.476	1.209	0.770
Corn	98.07	0.502	0.880	0.376
	122.58	0.540	0.948	0.378
	147.10	0.633	1.155	0.412
	171.62	0.625	1.229	0.483

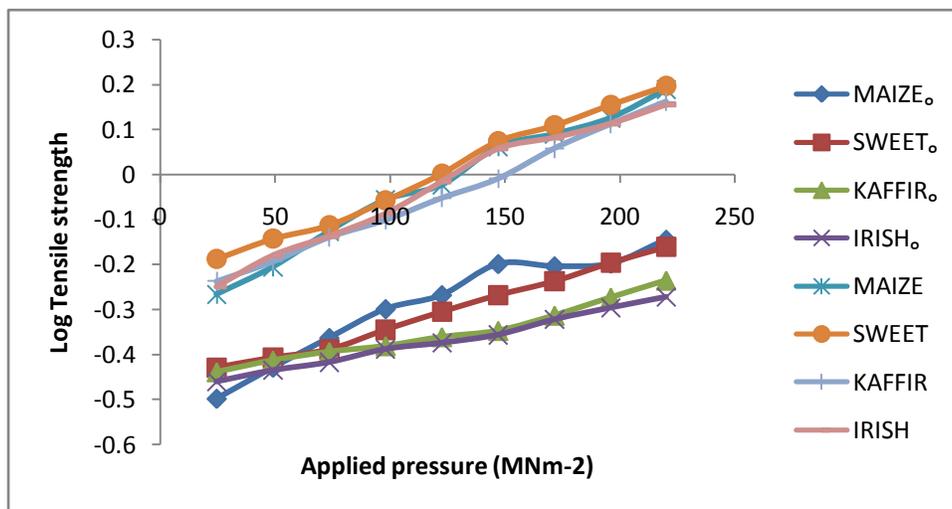


Figure 1: plots of log tensile strength Vs applied pressure of tablets with and without holes at constant (98.07MNm⁻²) applied pressure

Table 2: The results of tensile strength and Brittle Fracture Index of Paracetamol tablet formulation with increasing binder concentration and constant compression pressure(98.07 MNm⁻²)

Starch	Conc.(W/V)	T _o	T	BFI
Kaffir	2.5	0.344	0.642	0.492
	5.0	0.416	0.792	0.516
	7.5	0.518	0.900	0.457
	10.0	0.557	1.123	0.605
Sweet	2.5	0.369	0.686	0.470
	5.0	0.452	0.875	0.515
	7.5	0.532	0.948	0.580
	10.0	0.588	1.311	0.634
Irish	2.5	0.332	0.608	0.505
	5.0	0.409	0.818	0.642
	7.5	0.492	0.891	0.453
	10.0	0.566	1.097	0.563
Corn	2.5	0.330	0.655	0.589
	5.0	0.502	0.880	0.378
	7.5	0.628	1.090	0.348
	10.0	0.595	1.157	0.501

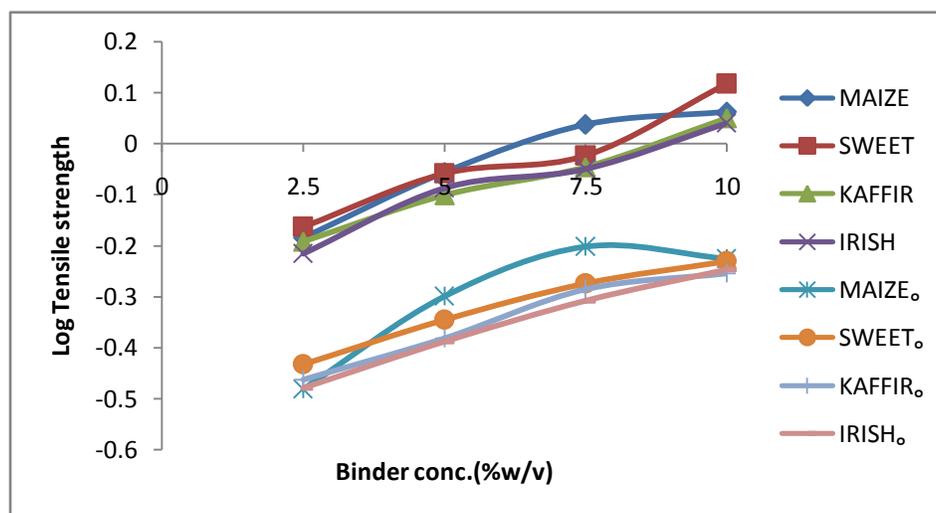


Figure 2: plots of log tensile strength Vs applied pressure of tablets with and without holes at constant (5%w/v) binder concentration.

The effects of increase in compression pressure or binder concentration on the porosity of tablets are presented in figures 3 and 4 respectively. The porosity of all the binders decreases with increase in compression pressure or binder concentration. The decrease in porosity as a result increased compression pressure and binder concentration could be responsible for increased tensile strengths. Tablets with central holes exhibited higher porosity than those without central hole.

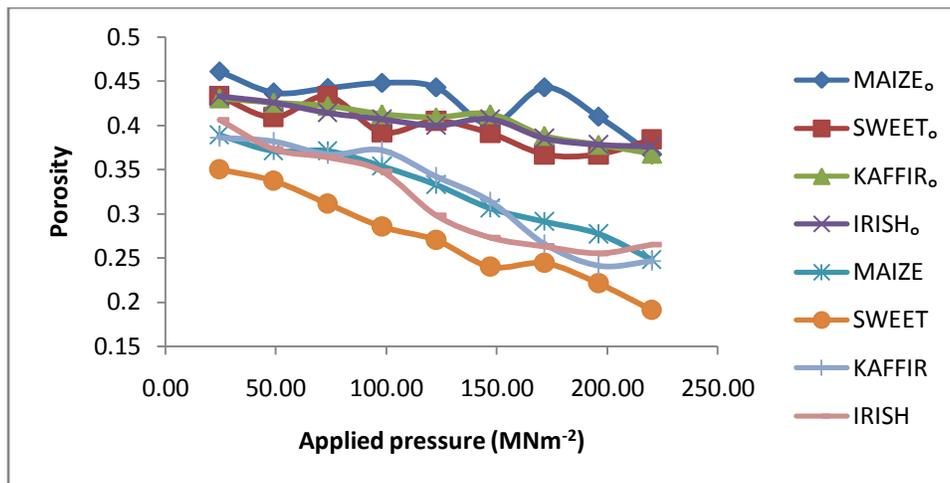


Figure 3:The effect of compression pressure on porosity of tablets produced with different binders.

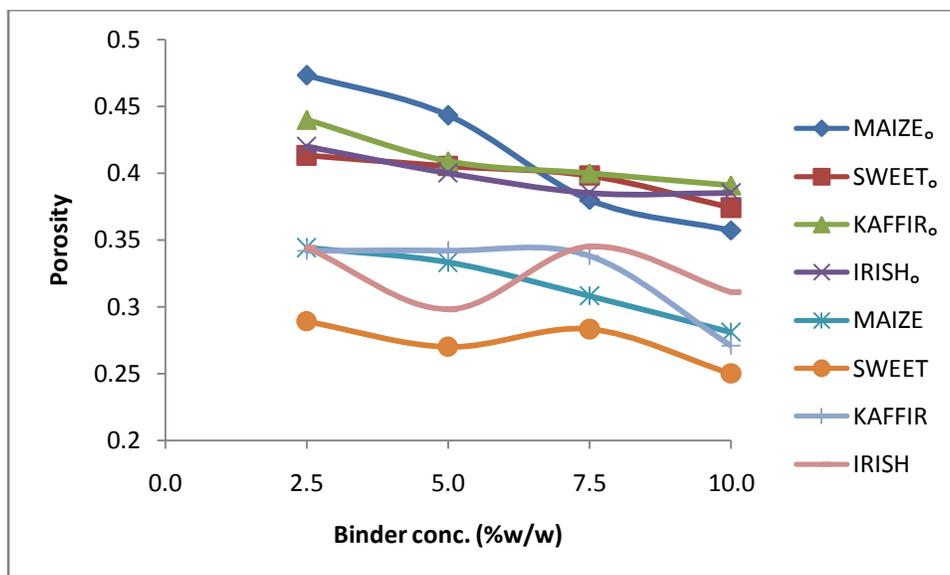


Figure 4:The effect of binder concentration on porosity of tablets produced at the same compression pressure.

The Heckel plots for a Paracetamol tablet formulation representative of 5% w/v of various starches are shown in figure 5. The mean yield value P_y was calculated from the portion of the plots showing linearity for all the formulation (between 24.52 to 220.25 MNm⁻²), intercept, A, was determined from the extrapolation of linear portion. The values of P_y , D_o , D_A and D_B for the formulations are presented in table 3, the D_o values which represents the degree of initial packing in the die as a result of die filling for the formulations increased with increased in starch concentration with Kaffir potato starch exhibiting the highest D_o . The ranking of the D_o value for the formulation was Kaffir > Sweet potato > Irish > Corn. This shows that formulation containing corn starch exhibited the lowest values.

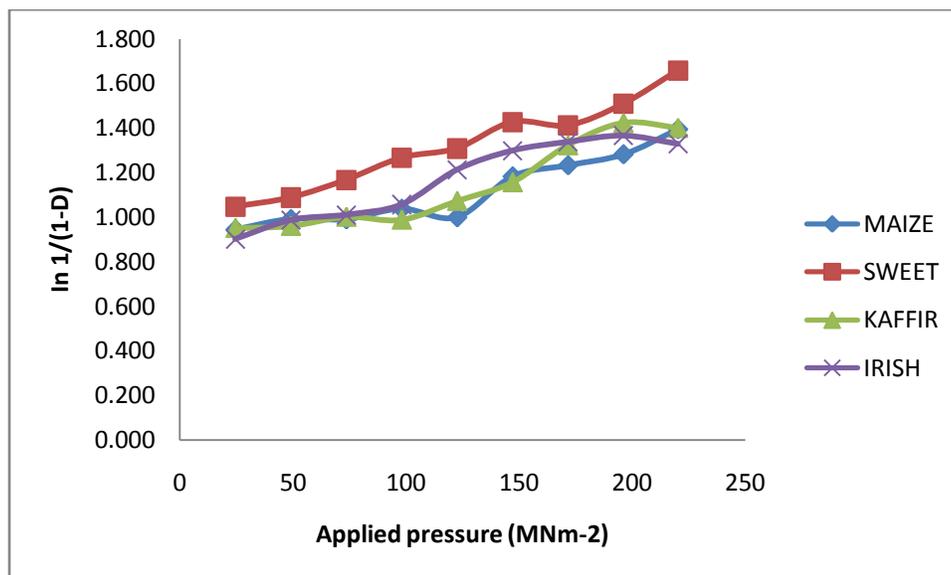


Figure 5: Plots of $\ln 1/(1-D)$ Vs applied pressure at binder concentration 5%w/v.

Table 3: Result of effect of varying binder concentration on Heckel and Kawakita constants in Paracetamol tablet formulations.

Starch	Conc.	Heckel plots				Kawakita plots	
		D_0	P_y	D_A	D_B	P_k	D_I
Kaffir	0	0.217	196.71	0.735	0.518	4.001	0.486
	2.5	0.223	347.81	0.818	0.595	2.627	0.482
	5.0	0.241	263.68	0.802	0.561	2.195	0.496
	7.5	0.279	223.61	0.794	0.515	2.008	0.512
	10.0	0.269	210.01	0.778	0.509	1.954	0.538
Sweet	2.5	0.232	388.48	0.825	0.593	2.545	0.483
	5.0	0.249	289.74	0.813	0.564	2.472	0.532
	7.5	0.256	236.77	0.812	0.556	2.306	0.549
	10.0	0.264	230.11	0.788	0.524	2.122	0.561
Irish	2.5	0.225	323.57	0.837	0.613	2.367	0.413
	5.0	0.236	271.06	0.833	0.597	2.105	0.419
	7.5	0.263	217.24	0.83	0.567	1.996	0.428
	10.0	0.257	188.74	0.826	0.569	1.948	0.447
Corn	2.5	0.226	307.11	0.81	0.584	2.107	0.452
	5.0	0.231	237.42	0.802	0.571	2.014	0.461
	7.5	0.247	215.88	0.798	0.551	1.984	0.498
	10.0	0.255	189.9	0.793	0.538	1.942	0.523

The degree of packing at zero and low pressure D_A decreased with increased in concentration of starch binder. The ranking was Irish > Sweet > Kaffir > Corn. Formulation containing Irish potato starch thus, exhibited high degree of total packing in the die at zero and low pressures while formulations containing corn starch had the lowest values.

The D_B values, which represents the particle rearrangement phase in the early compression stages and indicates the extent of fragmentation of granules, although fragmentation may occur

concurrently with plastic or elastic deformation of constituent granule²³. The values (D_B) are seen to decrease with increased in the concentration of starch for all formulation. This also indicates that fragmentation of granules decreased with increased in the concentration of binders (Starch). The ranking of the D_B value was Irish > Sweet > kaffir > Corn. Thus, Irish potato starch and corn starch exhibiting highest and lowest values of D_B respectively.

The P_y is inversely related to the ability of the fragmentation to deform plastically under pressure. The values of P_y decreased with increased in starch concentration for all the formulations. The ranking of P_y values was Sweet > Kaffir > Irish > Corn. This showed that formulation with Corn starch and sweet potato starch exhibited lowest and highest onset of plastic deformation respectively.

The Kawakita plots for Paracetamol formulations containing 5% w/v of all the starches are shown in figure 6. A near linear relationship between P/C and applied pressure was obtained for all the starches values of a and b were obtained from the slope and intercept of the plots respectively as explained earlier.

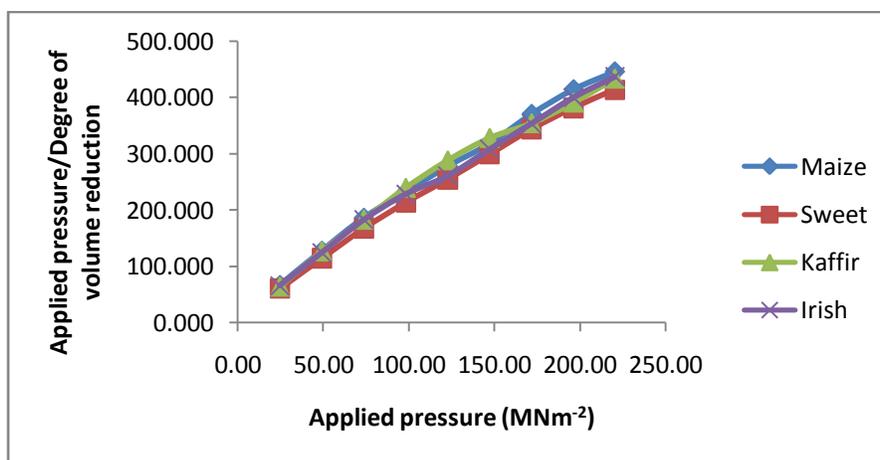


Figure 6: Kawakita plots of corn, kaffir, sweet and Irish potato starches at binder concentration of 5%w/v

The initial relative density D_I was obtained from the values of $1-a$ while P_k values, which is an inverse measure of the amount of plastic deformation occurring during compression²³ was obtained from the reciprocal values of b and are also shown on table 3.

The D_I increased with increase in binder concentration for all the formulations. The ranking was generally sweet > Kaffir > Corn > Irish.

Therefore formulation containing sweet potato starch exhibited the highest degree of packing with the application of small pressure or tapping. This is also in agreement with the work of other authors^{23,24}.

The value of P_k was observed to decreased with increased in binder concentration for all the formulations, the ranking of values of P_k was generally Kaffir > Irish > Sweet > Corn. Kaffir starch formulation exhibited highest amount of total plastic deformation and least observed in corn starch formulation.

CONCLUSION

Formulation with sweet potato starch produces tablets with strongest mechanical strength, while corn starch BP showed better BFI. Therefore, the potato starches can be employed interchangeably with corn starch BP in formulation of tablets.

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