



Survey the Crystal Type and Physiochemical properties of Isolated Starch Form Some Iraqi and Imported Genotypes of Corn (*Zea Mays*) Cultivated in Sulaimani-Iraq

Maki Mohammed Ahmad Tarkhani^{1*} and Muhammad Wajeeh M. Saeed Zainulabidden²

¹Biotechnology and Crop Sciences Department, Agricultural Engineering Sciences College, University of Sulaimani, Sulaimani, Iraq.

²Food Science and Quality Control Department, Agricultural Engineering Sciences College, University of Sulaimani, Sulaimani, Iraq.

*Corresponding author e-mail: Email: maki.ahmad@univsul.edu.iq

<https://doi.org/10.59658/jkas.v10i3.1243>

Received:

Aug. 2, 2023

Accepted:

Aug. 22, 2023

Published:

Sept. 10, 2023

Abstract

The objective of this study is to conduct a survey comparing ten different genotypes of corn. (*zea mays* L.) cultivated in Qlyassan research station of College of Agricultural Engineering Science, were to find out the type of crystalline form of starch extracted from studied corn genotypes. There were significant differences between the studied corn genotypes in the chemical composition, especially amylose and amylopectin, in which the amylose range was between 19.935 and 31.095 there were also differences in the mean sizes of the starch granules which was 12.795 to 15.915 μ m, as well as for Gel consistency, despite of significant differences between corn genotypes starch, all genotypes undergo to soft gel consistency, but all of these were not have affects the type of amyloid crystal, which was type A.

Keywords: cornstarch, amylose, amylopectin, granule size, Gel consistency, X-Ray diffraction.

Introduction

Understanding starch crystallinity is a crucial factor in determining the type of starch and its physicochemical properties and the effect of these properties on manufacturing quality, in addition to the close relationship between starch crystallinity and its resistance to digestive enzymes such as Alfa amylase enzyme. This is what made a lot of research tend to modify the starch associated with changing its crystal form, whether by thermal, physical, chemical or enzymatic methods [1]. Cultivation of corn in Iraq constitutes an important economic pillar for the state and farmers. In 2020, maize production for Iraq was 365 thousand tonnes [2]. Finding new ways to manufacture corn will greatly help in increasing the cultivation of this bio-product. The identification of the type of starch crystal in corn genotypes grown in Iraq did not receive clear interest in the research adopted in Iraq on corn starch The primary objective of this research is

to investigate the crystal models of starch extracted from several of the most widely cultivated varieties in Iraq.

Materials and Methods

Samples collection

Ten corn genotypes were used in this study that obtained from different sources Table (1). Six of these genotypes which are ZP.434*A, ZP.434*B, MSI*B, Dhqan, Corpeto and Dracma has been cultivated in 2019 in Qlyassan research station of College of Agricultural Engineering Science at Sulaimani University for grain production.

Table (1): The corn genotypes used in this study and their sources

	Genotypes	Sources
1	Sara	General Commission for Iraqi Agricultural Research - Field Crops Research Section
2	Al Maha	
3	Fajr-1	
4	Bagdad-3	
5	ZP.434*A	Ministry of Agriculture and water Resource in Kurdistan region-Iraq
6	ZP.434*B	
7	MSI*B	
8	Dhqan	University of Kurdistan, Sianandaj, Iran
9	Corpeto	Syngenta Italia S.p.A company
10	Dracma	

Starch isolation

In this research, approximately 1 kg of corn grains was ground using a laboratory blender (zeveloo, 150 g, stainless steel, 1500 w) to obtain corn flour. After sieving the flour, it mixed with water to make a ball of dough. Subsequently, the dough was placed in a pot, having small amounts of water left resting for an hour. The was done to prepare the dough for analysis related to starch crystallinity in the corn genotypes being studied. putting the slurry through filtration of 75µm mesh sieve, to distinct the non-starchy residue, then centrifuged at 4000 rpm for 20 minutes. The top protein rich layer in grey color removed by spatula. Amount water was added to re-suspend the sample, and centrifugation was done again for 20 min, this process was repeated several times until the obtain starch. The crude starch was refined by using proteinase-k solution (18000 units/mg) to erase filter as much proteins as possible. The purified starch was dried for 24 h at 40°C [3, 4].

Chemical composition

Moisture, for each starch sample was determined according to [5], while protein, lipid, and ash were set on [6]. Total carbohydrate was calculated by the differences also according to [7].

Amylose content

Estimation of amylose content of isolated starch was conducted by using the method of [8] with some modifications, then the amylose content was found by using the following equation;

$$\text{Amylose content (\%)} = (85.24 \times A) - 13.19$$

$$\text{Amylopectin (\%)} = 100 - \% \text{ Amylose}$$

Determination of starch granule size

Starch samples' granule sizes were measured using a procedure expressed by [9] with some modifications. After measuring the granule sizes of starch it was formulated by using the mean diameters of the granules then multiplied by a factor of 2.5 μm to determine their actual sizes, (which was formulated previously by using the parallax obtained between platform micrometer and the calibrations of the eyepiece).

Gel consistency

The method of [10] was used to know the gel consistency of corn genotypes starch.

X-ray diffraction

The crystalline structure of the starches was taken through X-Ray Diffraction (XRD) analysis that described by [11] with some modifications by using a Bruker D8 advance X-ray diffractometer.

Statistical Analysis

The data's obtained were faced to one-way analysis of variables (ANOVA) at $P < 0.05$. The analysis was performed and the results were separated using the Multiple Range Duncan's test using XLSTAT, version (2016. .02.28451) statistical software for the analytical data, mean values and standard deviation.

Results and Discussion

Chemical composition

The chemical components of the starch samples which analyzed in this study displayed on Table (1). Moisture content ranged between (9.253 to 12.245%). According to statistical analysis of the results, moisture content is less than the permissible value which is 13%, according to the (Iraqi Standard Specifications, No. 1209 of year 1988) for imported starch. Therefore, the lower content of moisture diminishes the danger of

microbial growth while being stored, this was mentioned by [12]. The samples' protein content ranged from (0.770 to 1.540 %). Significant differences in the protein composition of five samples of starch were shown by statistical analysis of the results [13]. Oil content of these starch samples ranged between (0.309 to 0.664 %), Although it is unacceptable, but the reason might be due to the method of extraction, like that, it has been reported by [14], the high purity of separated starches is indicated by low fat and protein levels. Ash content ranged from (0.122 to 0.214 %), These results were in a good agreement with the results which reported by [15, 16]. So, the starches which obtained from these genotypes might be categorized and utilized as normal corn starches [17, 18]. The higher values of the chemical components may be attribute to their responses to extraction treatments due to their differences in their structure or the physical state surrounding or the inner of starch granules.

Table (1): Proximate analysis of cornstarch extracted from studied corn genotypes

Genotypes	Ash content %	Moisture content %	Oil content %	Protein content %	Carbohydrate content %
Sara	0.126±0.001 ^h	9.812±0.001 ^h	0.453±0.001 ^h	1.433±0.058 ^b	88.176±0.055 ^c
Al Maha	0.194±0.001 ^b	9.253±0.001 ^j	0.491±0.001 ^f	1.210±0.010 ^d	88.852±0.009 ^a
Fajr-1	0.146±0.001 ^e	10.212±0.001 ^d	0.461±0.001 ^g	1.433±0.015 ^b	87.748±0.013 ^f
Bagdad-3	0.141±0.001 ^f	10.283±0.001 ^c	0.592±0.002 ^c	1.250±0.010 ^c	87.734±0.006 ^f
ZP.434*A	0.122±0.001 ⁱ	12.245±0.001 ^a	0.432±0.001 ⁱ	1.150±0.010 ^e	86.051±0.010 ^h
ZP.434*B	0.214±0.001 ^a	10.152±0.001 ^e	0.612±0.002 ^b	1.540±0.010 ^a	87.482±0.009 ^g
MSI*B	0.136±0.001 ^g	9.644±0.001 ⁱ	0.664±0.001 ^a	1.433±0.015 ^b	88.123±0.014 ^d
Dhqan	0.172±0.001 ^d	9.953±0.001 ^g	0.584±0.002 ^d	1.150±0.010 ^e	88.141±0.006 ^d
Corpeto	0.176±0.001 ^c	10.563±0.001 ^b	0.309±0.002 ^j	1.143±0.015 ^e	87.809±0.015 ^e
Dracma	0.136±0.001 ^g	10.143±0.001 ^f	0.507±0.002 ^e	0.770±0.010 ^f	88.444±0.011 ^b

Means with a different small letter in the same column are significantly different (P<0.05)

Amylose and Amylopectin content

The results in Table (2) showed that there was a significant difference between corn varieties starches in their content of amylose and amylopectin. The amylose content varied among all of the variety's starches. The maximum value of amylose was recorded for ZP.434*B which was (31.095%) and minimum value was recorded by Sara which was (19.935%). These values of amylose percentages are in accordance with [19] who found that the range of amylose was between 18-35% for studied normal starch. The percentage of amylose to amylopectin plays the most important role in effect of all physical and biochemical properties which the resistant starch formation completely depends on this ratio more than other studied measurements.

Table (2): Some physiochemical properties of cornstarch extracted from studied corn genotypes

Genotypes	Amylose %	Amylopectin %	Granule size μm
Sara	19.935 ±0.010 ^j	80.078 ± 0.004 ^a	13.126±0.001 ^h

Al Maha	24.113 ±0.012 ^g	75.897 ± 0.001 ^d	13.863±0.001 ^c
Fajr-1	20.140 ±0.002 ⁱ	79.864 ± 0.002 ^b	13.730±0.036 ^d
Bagdad-3	20.271 ±0.005 ^h	79.735 ± 0.001 ^c	12.795±0.001 ⁱ
ZP.434*A	26.669 ±0.005 ^e	73.344 ± 0.004 ^g	13.360±0.010 ^f
ZP.434*B	31.99 ±0.015 ^a	68.910 ± 0.002 ^j	13.317±0.001 ^g
MSI*B	29.531 ±0.025 ^d	70.486 ± 0.001 ^h	15.915±0.001 ^a
Dhqan	30.192 ±0.002 ^b	73.811 ± 0.002 ^e	13.520±0.010 ^e
Corpeto	29.944 ±0.003 ^c	70.061 ± 0.002 ⁱ	14.415±0.001 ^b
Dracma	26.319 ±0.002 ^f	73.681 ±0.001 ^g	13.370±0.010 ^f

Means with a different small letter in the same column are significantly different (P<0.05)

The importance of estimating the starch content of amylose and amylopectin comes from the fact that these two factors are the determinants of the starch crystal model, whether by the ratio of one to the other or by the type of structures that each of them will be, whether in the natural state or during thermal and chemical treatments. It is known that increasing the percentage of amylopectin to 99% will produce a waxy starch, and the crystal pattern will be different from that of natural starch [20, 21].

Granule size means

There are differences among starch granules size that shown in Table (2). An average granule size mean's varied from (15.915 µm) for MSI*B to (12.795 µm) for Bagdad-3. Variation in starch granule size is influenced by both genetic composition and environmental conditions, which significantly impact the quality of starch [22, 23]. Additionally, the shape and size of granules play a crucial role in the starch extraction industry, as they determine the mesh size of sieves used for application and purification [24]. Furthermore, starch granule size has a direct effect on water absorption, solubility, and swelling power [25]. Smaller starch granules, as mentioned by [26], tend to exhibit greater solubility and an increased capacity to absorb water.

Gel consistency (mm)

Deferent letters in the figure refer to significant differences (p < 0.05) of gel consistency values among various starches of corn genotypes that observed in Figure (1), which were recorded from 67 to 121 mm, despite of different values recorded in the results, all cultivars undergo to soft gel consistency and viscosity [10] which they reported that the distance of gel flow (long which is mean soft gel and short means hard gel) inversely correlated with the amylose content. Gel consistency is a measure of how likely gelatinized starch is to retrograde upon cooling. Amylose, with its linear form, tends to retrograde more readily than amylopectin within the starch. This difference arises from amylose's ability to readily form hydrogen bonds between molecules due to its straight chain structure. Starch gel formation primarily relies on swollen starch

granules that trap water in the granule's network [27]. As a result, the mechanical strength of starch gels is mainly influenced by the composition, granule characteristics, and molecular interactions with water [28].

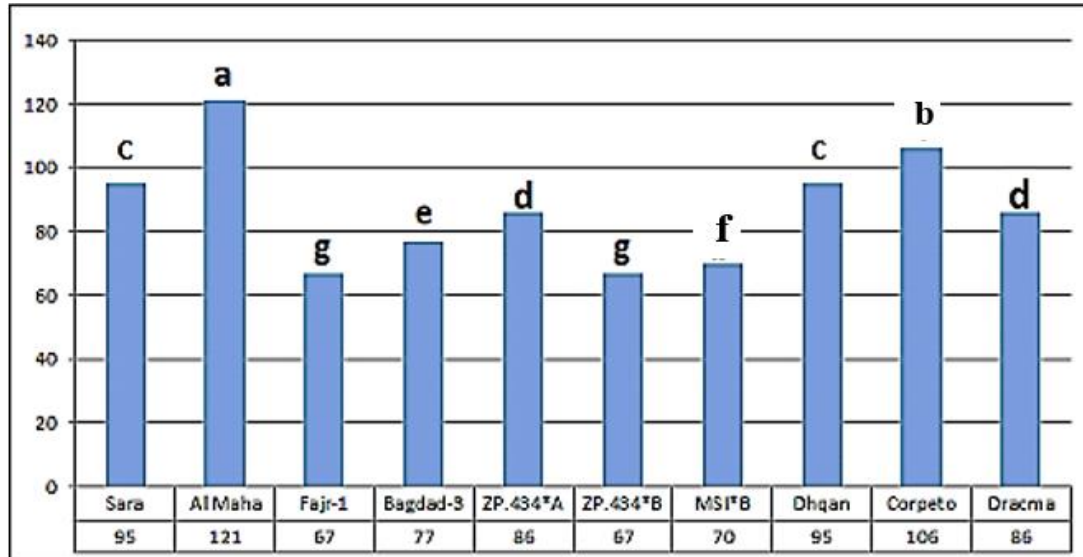


Figure (1): Gel consistency of ten cornstarch genotypes under the study. Means with a different small letter are significantly different ($P < 0.05$)

X-Ray diffraction

Figure (2) shows the X-ray diffract graph of starch samples, revealing a consistent A-type crystalline pattern across all starch samples. The pattern exhibits high-intensity peaks at specific Bragg's angles (2θ) approximately at 15.15° , 17.39° , 18.11° , and 23.20° . This A-type crystalline structure is similar to that observed in other cereal starches, indicating the semi-crystalline nature of corn starches, as reported by [29]. Previous studies on corn starch X-ray diffraction, including [30], also confirmed the presence of the A-type pattern with strong reflections at 2θ around 15° , 18° , and 23° . The crystalline structure of starch indicates the absence of organized helices, and it is influenced by factors such as the chain length of amylose and amylopectin, as well as the arrangement of double helix chains, as stated by [31, 32]. Differences in the type-A pattern are attributed to the chain length of amylopectin. Hizukuri [33] identified that in type-A crystalline pattern, amylopectin consists of a higher proportion of short chains (below DP 19), whereas in Type-B crystalline pattern, it comprises a higher proportion of longer chains (DP = 20 to 37). However, despite the amylopectin percentage ranging from 68.910 ± 0.002 to 80.078 ± 0.004 , it does not have any significant effect on the crystallinity model of the tested cornstarch.

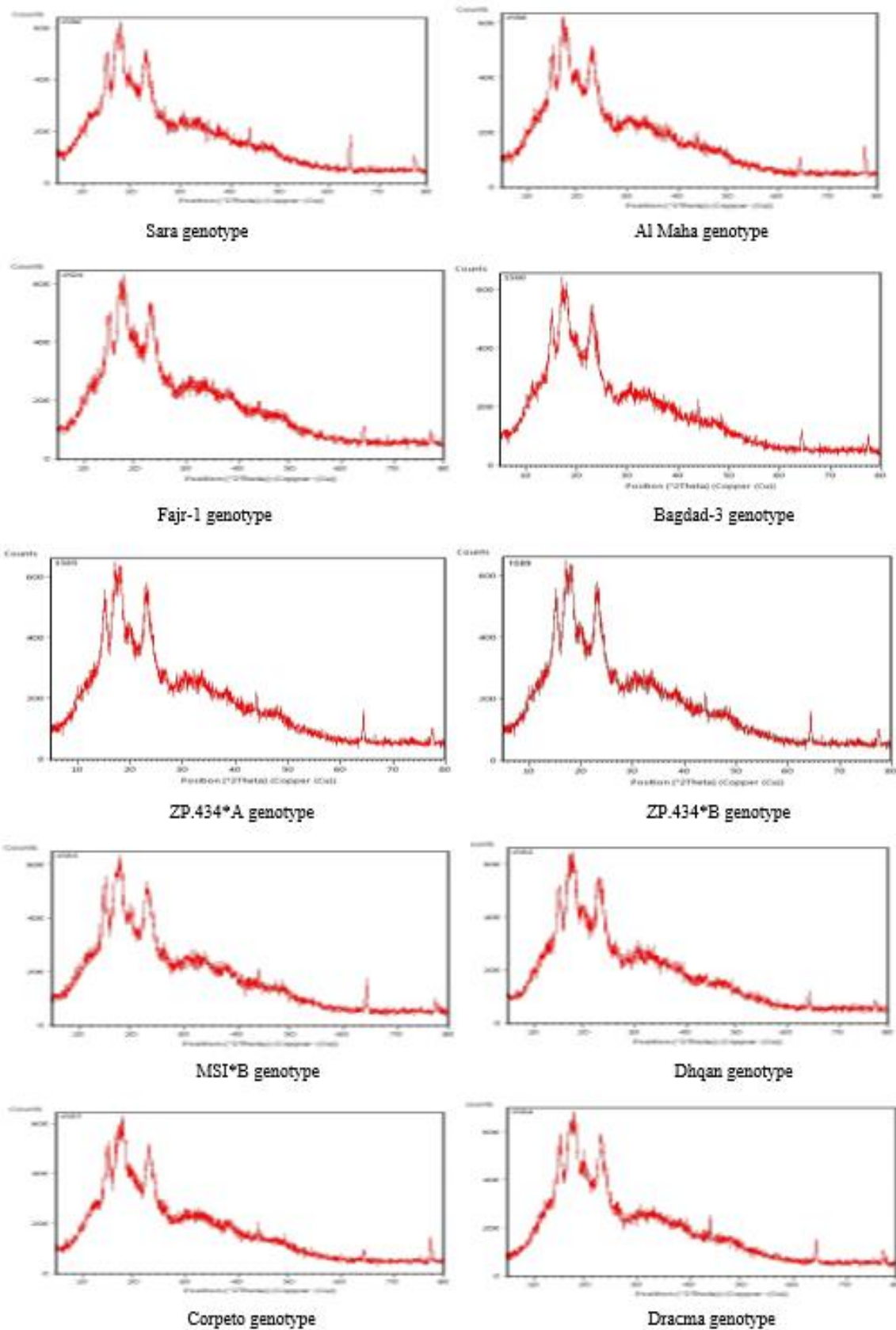


Figure (2): X-ray diffraction patterns of starches isolated from ten corn genotypes



The research findings indicate that the variations in starch granule size and the proportions of amylose and amylopectin observed in this study did not have a notable impact on the starch crystal model. Surprisingly, even the starch extraction processes employed did not significantly influence the type of crystal formed, despite some prior research suggesting that extraction processes might affect crystal type [20]. Consequently, it can be inferred that the genetic factor plays a more crucial role in determining the crystal form compared to other factors investigated in this study.

References

- 1) Guo, J.: Wang, Z.: Qu, L.: Hao, D. and Lu, D.(2023). Comparison of the physicochemical properties of starches from maize reciprocal F1 hybrids and their parental lines. *Food Chemistry*: X, p.100561.
- 2) Knoema, (2023). World Data Atlas Iraq Topics Agriculture Crops Production, [Https://Knoema.Com](https://Knoema.Com)
- 3) Grant, L.A.(1998). Effects of starch isolation, drying, and grinding techniques on its gelatinization and retrogradation properties. *Cereal Chemistry*, 75(5): pp.590-594.
- 4) Sandhu, K.S., Singh, N. and Malhi, N.S.(2005). Physicochemical and thermal properties of starches separated from corn produced from crosses of two germ pools. *Food Chemistry*, 89(4): pp.541-548.
- 5) AACC, (2000). Approved Method of American Association of Cereal Chemists. St. Paul, MN.: AACC international.
- 6) AOAC, (2012). Official methods of analysis of AOAC international, 2012, Gaithersburg, Maryland, USA: 19th edition. AOAC International.
- 7) Moraes, É.A.; Dantas, M.I.D.S.; Morais, D.D.C.; Silva, C.O.D.; Castro, F.A.F.D.; Martino, H.S.D. and Ribeiro, S.M.R.(2010). Sensory evaluation and nutritional value of cakes prepared with whole flaxseed flour. *Food Science and Technology*, (30) pp.974-979.
- 8) Williams, P.C.; Kuzina, F.D. and Hlynka, I.(1970). Rapid colorimetric procedure for estimating the amylose content of starches and flours. *Cereal chemistry*.
- 9) Amoo, A.R.N.; Dufie, W.M.F. and Ibok, O.(2014). Physicochemical and pasting properties of starch extracted from four yam varieties. *Journal of Food and Nutrition Sciences*, 2(6): pp.262-269.
- 10) Cagampang, G.B.; Perez, C.M. and Juliano, B.O.(1973). A gel consistency test for eating quality of rice. *Journal of the Science of Food and Agriculture*, 24(12): pp.1589-1594.



- 11) Huang, J.; Schols, H.A.; van Soest, J.J.; Jin, Z.; Sulmann, E. and Voragen, A.G. (2007). Physicochemical properties and amylopectin chain profiles of cowpea, chickpea and yellow pea starches. *Food Chemistry*, 101(4): pp.1338-1345.
- 12) Alcázar-Alay, S.C. and Meireles, M.A.A., 2015. Physicochemical properties, modifications and applications of starches from different botanical sources. *Food Science and Technology*, (35): pp.215-236.
- 13) Bustillos-Rodríguez, J.C.; Tirado-Gallegos, J.M.; Ordonez-Garcia, M.; Zamudio-Flores, P.B.; Ornelas-Paz, J.D.J.; Acosta-Muñiz, C.H.; Gallegos-Morales, G.; Páramo-Calderón, D.E. And Rios-Velasco, C.(2018). Physicochemical, Thermal and Rheological Properties of Three Native Corn Starches. *Food Science and Technology*, (39): Pp.149-157.
- 14) Tirado-Gallegos, J.M.; Zamudio-Flores, P.B.; Ornelas-Paz, J.D.J.; Rios-Velasco, C.; Acosta-Muñiz, C.H.; Gutiérrez-Meraz, F.; Islas-Hernández, J.J. and Salgado-Delgado, R. (2016). Efecto del método de aislamiento y el estado de madurez en las propiedades fisicoquímicas, estructurales y reológicas de almidón de manzana. *Revista Mexicana de Ingeniería Química*, 15(2): pp.391-408.
- 15) Abdalla, A.A.; Ahmed, U.M.; Ahmed, A.R.; El Tinay, A.H. and Ibrahim, K.A. (2009). Physicochemical characterization of traditionally extracted pearl millet starch (Jir). *J Appl Sci Res*, 5(11): pp.2016-2027.
- 16) Ali, H.A.E.A., (2008). Evaluation of Six Corn (*Zea mays* L.) Hybrids for wet-milling and starch quality (Doctoral dissertation, M. Sc. Thesis. University of Khartoum. Sudan).
- 17) Aparicio-Saguilán, A.; Méndez-Montevalvo, G.; Solorza-Feria, J. and Bello-Pérez, L.A.(2006). Thermal and viscoelastic properties of starch gels from maize varieties. *Journal of the Science of Food and Agriculture*, 86(7): pp.1078-1086.
- 18) Hardacre, A.K. and Clark, S.M., (2006). The effect of hybrid and growing environment on the rheological properties of starch and flour from maize (*Zea mays* L.) grain dried at four temperatures. *International journal of food science & technology*, 41, pp.144-150.
- 19) Wu, X.; Zhao, R.; Wang, D.; Bean, S.R.; Seib, P.A.; Tuinstra, M.R.; Campbell, M. and O'brien, A. (2006). Effects of amylose, corn protein, and corn fiber contents on production of ethanol from starch-rich media. *Cereal chemistry*, 83(5): pp.569-575.
- 20) Bertoft, E. (2017). Understanding starch structure: Recent progress. *Agronomy*, 7(3): p.56.



- 21) Dome, K.; Podgorbunskikh, E.; Bychkov, A. and Lomovsky, O. (2020). Changes in the crystallinity degree of starch having different types of crystal structure after mechanical pretreatment. *Polymers*, 12(3): p.641.
- 22) Li, W.Y.; Yan, S.H.; Yin, Y.P.; Yong, L.I.; Liang, T.B.; Feng, G.U.; Dai, Z.M. and Wang, Z.L.(2008). Comparison of starch granule size distribution between hard and soft wheat cultivars in eastern China. *Agricultural Sciences in China*, 7(8): pp.907-914.
- 23) Singh, S.; Singh, N.; Isono, N. and Noda, T. (2010). Relationship of granule size distribution and amylopectin structure with pasting, thermal, and retrogradation properties in wheat starch. *Journal of agricultural and food chemistry*, 58(2): pp.1180-1188.
- 24) Leonel, M.; Sarmiento, S.B. and Cereda, M.P. (2003). New starches for the food industry: *Curcuma longa* and *Curcuma zedoaria*. *Carbohydrate Polymers*, 54(3): pp.385-388.
- 25) Hedayati, S.; Shahidi, F.; Koocheki, A.; Farahnaky, A. and Majzoobi, M. (2016). Functional properties of granular cold-water swelling maize starch: Effect of sucrose and glucose. *International Journal of Food Science & Technology*, 51(11): pp.2416-2423.
- 26) Agnes, A.C.; Felix, E.C. and Ugochukwu, N.T.(2017). Morphology, rheology and functional properties of starch from cassava, sweet potato and cocoyam. *Asi J Biol*, 3, pp.1-13.
- 27) Martin, C. and Smith, A. M. (1995). Starch biosynthesis. *The plant cell*, 7(7): 971.
- 28) Nadiha, M.N.; Fazilah, A.; Bhat, R. and Karim, A.A. (2010). Comparative susceptibilities of sago, potato and corn starches to alkali treatment. *Food Chemistry*, 121(4): pp.1053-1059.
- 29) Suma P, F. and Urooj, A. (2015). Isolation and characterization of starch from pearl millet (*Pennisetum typhoidium*) flours. *International Journal of Food Properties*, 18(12): pp.2675-2687.
- 30) López, O.V.; Zaritzky, N.E. and García, M.A.(2010). Physicochemical characterization of chemically modified corn starches related to rheological behavior, retrogradation and film forming capacity. *Journal of food engineering*, 100(1): pp.160-168.
- 31) Tester, R.F.; Karkalas, J. and Qui, X. (2004). Starch—composition, fine structure and architecture. *Journal of cereal science*, 39(2): pp.151-165.
- 32) Bertoft, E.; Piyachomkwan, K.; Chatakanonda, P. and Sriroth, K.(2008). Internal unit chain composition in amylopectins. *Carbohydrate Polymers*, 74(3): pp.527-543.



- 33) Hizukuri, S. (1986) . Polymodal distribution of the chain lengths of amylopectins, and its significance. Carbohydr. Res., 147, pp.342-347.