Talinum triangulare (Jacq.) Willd. mucilage and pectin in the formulation of ibuprofen microspheres

Olufunke Dorothy Akin-Ajani[A,C–F], Temiloluwa Mary Hassan[B,C,F], Oluwatoyin Adepeju Odeku[C,E,F]

Department of Pharmaceutics and Industrial Pharmacy, University of Ibadan, Nigeria

A – research concept and design; B – collection and/or assembly of data; C – data analysis and interpretation;
D – writing the article; E – critical revision of the article; F – final approval of the article

Polymers in Medicine, ISSN 0370-0747 (print), ISSN 2451-2699 (online) *Polim Med*. 2022;52(2):83–92

Address for correspondence
Olufunke Dorothy Akin-Ajani
E-mail: oakinajani@yahoo.com

Funding sources
None declared

Conflict of interest
None declared

Acknowledgements
The authors would like to thank the University of Potsdam, Golm Campus, Germany, for the use of their scanning electron microscope during O.D. Akin-Ajani and O.A. Odeku's research visit.

Received on July 8, 2022
Reviewed on July 13, 2022
Accepted on September 5, 2022

Published online on October 21, 2022

Abstract

Background. Mucilage and pectin are both natural polymers with the advantages of availability and biodegradability. Microspheres made from biodegradable polymers can break down naturally after performing their tasks.

Objectives. The study aimed to use mucilage and pectin from the leaves of *Talinum triangulare* (Jacq.) Willd. as polymer matrices for the formulation of microspheres, with ibuprofen as the model drug.

Materials and methods. Both polymers were examined under a microscope and evaluated using measurements of viscosity, density, flow properties, swelling power, elemental analysis, Fourier-transform infrared spectroscopy (FTIR), and the degree of esterification (DE) for pectin. The microspheres were prepared using the ionotropic gelation method and alginate:mucilage/pectin at ratios of 1:1 and 1:2. They were assessed for swellability, drug entrapment effectiveness and drug release profile.

Results. The mucilage particles were ovoid while pectin particles were irregularly shaped. Pectin had higher particle, bulk and tapped densities than mucilage, while mucilage had a higher swelling power and a better flow than pectin. *Talinum triangulare* pectin is a low-methoxyl pectin with a DE of 7.14%. The FTIR spectra showed no interaction between the polymers and ibuprofen. The surface morphology of the microspheres without ibuprofen was smooth, while those with ibuprofen revealed a spongy-like mesh. The swelling power of the microspheres was higher in phosphate buffer with a pH of 7.2 than in distilled water. The entrapment efficiency ranged within 39.57–60.43% w/w, with microspheres containing alginate:mucilage/pectin ratio of 1:1 having higher entrapment efficiency. Microspheres with polymer at a ratio of 1:1 provided a longer release (>2 h), while microspheres with polymer blend of 1:2 provided an immediate release of ibuprofen.

Conclusions. The polymers of *T. triangulare* could be used as matrices in microsphere formulations.

Key words: *Talinum triangulare*, microspheres, polymers, pectins, ibuprofen

Cite as
Akin-Ajani OD, Hassan TM, Odeku OA. *Talinum triangulare* (Jacq.) Willd. mucilage and pectin in the formulation of ibuprofen microspheres. *Polim Med*. 2022;52(2):83–92. doi:10.17219/pim/153520

DOI
10.17219/pim/153520

Introduction

Mucilage and pectin are natural polymers used in the pharmaceutical and food industries, with the advantages of chemical inertness and biodegradability.[1,2] While mucilages are polysaccharides that produce monosaccharides upon hydrolysis as well as sugars such as arabinose and galactose, they do not readily dissolve in water, but rather form slimy masses.[3] Pectin, on the other hand, is mostly formed from citrus fruit peels, but is also found in potato pulp and cocoa husk. Pectin is a ionic polysaccharide (heteropolysaccharide chains form esterified D-galacturonic acid (1,4-linked-D-galacturonic acid)) naturally occurring in terrestrial plant cell walls, that undergoes chain–chain association and produces hydrogels when divalent cations are introduced.[2,4–8] One crucial factor used to categorize the various forms of pectin is the degree of esterification (DE), or the proportion of carboxyl groups that are esterified and present in the structure of the pectin.[9] According to Giacomazza et al., high-methoxyl pectin (HMP) with DE > 50% is primarily used in the food industry as a thickening and gelling agent.[10] Low-methoxyl pectin (LMP), which is typically produced by the de-esterification of HMP, exhibits DE < 50%.[11]

Waterleaf, or *Talinum triangulare* (Jacq) Willd., belongs to the Portulacaceae family. Waterleaf is also known as talinum, Ceylon spinach, Philippine spinach, etc., and locally in Nigeria, as Efo Gbure (Yoruba), Mgbolodi (Igbo), Alenruwa (Hausa), and Ebe-dondon (Edo).[12] Talinum has about 40 known species and is particularly abundant in tropical Africa, USA and Mexico.[13] It is used as an ornamental plant in southern Asia and to make vegetable soup in the southern parts of Nigeria and other parts of West and Central Africa.[14,15] The average annual yield of talinum has continued to increase as more farmers have begun to plant it.[13,16] Talinum, like most vegetables, has a short life cycle and is highly perishable; the shoots may start withering within a few hours of harvesting. It has, however, been shown that the dried leaves retain most of their nutritive value, even when sun-dried.[13,17] *Talinum triangulare* has antitumor, anti-inflammatory, antioxidant, and tyrosinase-inhibitory properties.[18–23] The hypoglycemic and antianemic effects of *T. triangulare* are most pronounced in pregnant women and small children.[24,25] Waterleaf is also known to be a rich source of phenolic antioxidants, vitamin C, sugar, magnesium, phosphorus, etc.[26]

Mucilage and pectin from *T. triangulare* have been extracted to determine their quality and nutritional and antioxidant properties, as well as the effect of season on its production.[15,26,27] However, no studies have investigated *T. triangulare* mucilage and pectin in drug formulation. In the present study, mucilage and pectin extracted from the leaves of *T. triangulare* have been used as polymer matrices for the formulation of microspheres, with ibuprofen as the model drug.

Materials and methods

Materials

The materials used include: ibuprofen (a gift from Bond Chemicals Nig. Ltd., Awe, Nigeria), sodium alginate (S.D. Fine Chem, Mumbai, India), zinc chloride (QFC Fine Chem, Mumbai, India), acetone (CDH Fine Chemical, New Delhi, India), diethyl ether, phosphate buffer (VWR Chemicals, Leuven, Belgium), and waterleaf (*T. triangulare* locally harvested from farmlands around the University of Ibadan, Nigeria).

Extraction of mucilage from *Talinum triangulare*

Fresh leaves of *T. triangulare* were separated from the stalk, weighed and cleaned with distilled water. The juice was extracted manually with a muslin cloth, and the mucilage was precipitated with ethanol (96%). Before filtering, the precipitated mucilage was cleaned with diethyl ether. After drying in a hot air oven (Laboratory Oven TT-9083; Techmel & Techmel, Venaville, USA) at 50°C, the mucilage was milled, sieved with a 250-µm mesh sieve and stored in a dry container.[28]

Extraction of pectin from *Talinum triangulare*

The dried leaves of *T. triangulare* (150 g) were put in a 2-liter beaker holding 500 mL of distilled water, and the mixture was allowed to boil for 45 min. The mixture was filtered using a muslin cloth, and 200 mL of 95% acetone was added to the filtrate in aliquots with continuous stirring to allow the pectin to precipitate.[29]

Particle size and morphology determination

Using an optical microscope (model 312545; Olympus Corp., Tokyo, Japan), the mucilage and pectin particle sizes of *T. triangulare* were determined. Under the microscope, the diameters of 100 different particles were measured to calculate the mean projected diameter in meters. The Motic Software (Motic MC2000 Image Capture Module; Motic China Group Co., Ltd., Xiamen, China) was used to take photomicrographs.

Density determinations

The bulk and tapped densities of the polymers were calculated as described previously by Ajala et al.[28] Particle densities of the polymers were determined with the liquid pycnometer method using xylene as the displacement fluid.

Hausner ratio and Carr's index

Hausner ratio was calculated by dividing the initial bulk volume by the tapped volume. The tapped volume was determined by applying 100 taps at a standardized rate of 38 taps per min to 5 g of polymer in a graduated cylinder.[30]

Equation 1 was used to calculate the Carr's index[31]:

$$\text{Carr's index (\%)} = \frac{\text{(tapped density} - \text{bulk density)}}{\text{tapped density}} \times 100 \quad (1)$$

Angle of repose

The polymer (5 g) was poured through a funnel into an open-ended cylinder positioned on a cone with a 2.8-cm diameter, and the cylinder was gently lifted vertically while the powder formed a mound. The angle of repose was calculated using the height (h) and radius (r) measurements (Equation 2):

$$\tan(\theta) = \frac{h}{r} \quad (2)$$

Swelling power

The polymer (0.5 g) was put into a measuring cylinder with a capacity of 10 mL, and the heights were recorded (h1). The polymer was mixed with phosphate buffer or distilled water to the 10 mL mark, and the resulting slurry was stirred for 5 min. The sedimentation height (h2) was measured after letting the suspension sit for 24 h. Next, the swelling power was determined[30] (Equation 3):

$$\text{swelling power} = \frac{v2}{v1} \quad (3)$$

where v1 and v2 are volumes derived from h1 and h2, respectively.

Characterization of pectin

The Ranganna's method was used to compute the equivalent weight of pectin.[32] In a 250-mL conical flask, pectin (0.5 g), ethanol (5 mL), sodium chloride (1 g), and distilled water (100 mL) were combined. This was titrated against 0.1 N NaOH using phenol red as an indicator. The endpoint was indicated by a change in color to purple. The equivalent weight of pectin was calculated using Equation 4, and the methoxyl content was determined using the neutralized solution[2]:

$$\text{equivalent weight} = \frac{\text{weight of sample} \times 1000}{\text{volume of alkali} \times \text{normality of alkali}} \quad (4)$$

The neutralized solution produced by the titration with equivalent weight was mixed with sodium hydroxide (25 mL of 0.25 N). After thoroughly stirring the mixture, it was allowed to stand at room temperature for 30 min. The mixture was then titrated against 0.1 N NaOH using 25 mL of 0.25-N hydrochloric acid.[32] Equation 5 was used to determine the methoxyl content of pectin[2]:

$$\text{methoxyl content (\%)} = \frac{\text{volume of alkali} \times \text{normality of alkali} \times 3.1}{\text{weight of sample}} \quad (5)$$

Equation 6 was used to calculate the total anhydrouronic acid (AUA) of pectin[2]:

$$\text{AUA (\%)} = \frac{176 \times 0.1z \times 100}{w \times 1000} + \frac{176 \times 0.1y \times 100}{w \times 1000} \quad (6),$$

where the molecular weight of 1 unit of AUA is 176 g, z is the titer volume (mL) of NaOH is determined by equivalent weight, and y is the titer volume (mL) of NaOH is determined by methoxyl content determination, while w is the weight of the pectin sample.

The degree of esterification was calculated from the percentage of methoxyl content (MeO) (Equation 5) and % AUA (Equation 6) using Equation 7:

$$\text{DE (\%)} = \frac{176 \times \%\ \text{MeO}}{31 \times \%\ \text{AUA}} \times 100 \quad (7)$$

An atomic absorption spectrophotometer (AAS, Model 2500; Torontech Inc., Toronto, Canada) was used to evaluate the pectin and mucilage for 10 elements.[33]

The Fourier-transform infrared spectroscopy (FTIR) spectra of dried powders of mucilage, pectin, alginate, and ibuprofen, as well as their mixes formed in potassium bromide (KBr) discs, were determined using an FTIR system (Spectrum BX 273; PerkinElmer, Waltham, USA), with a scanning range of 350–4400 cm^{-1}.

The viscosity of mucilage and pectin blends with sodium alginate was evaluated using a viscometer (model RVVDV-II +P; Brookfield Engineering Laboratories Inc., Middleboro, USA) with a spindle size of 4 at 50 rpm and 100 rpm.

Preparation of microspheres

Ibuprofen-loaded microspheres were created using the ionotropic gelation technique. Different batches of sodium alginate alone, as well as the polymer blends of different concentrations of alginate with mucilage and alginate with pectin in ratios 1:1 and 1:2, were prepared. Ibuprofen (1 g) was incorporated into various blends at a polymer-to-drug ratio of 2:1. The microspheres were prepared with 10% w/v zinc chloride as the crosslinking agent. The polymer mixture was extruded using a 21-G needle and a 5-mL syringe at a dropping rate of 2 mL/min and a stirring speed of 300 rpm. To begin the curing process, the microspheres were immersed in zinc chloride for 10 min. Then, they were filtered, rinsed 3 times with distilled water, dried at ambient temperature for 24 h, and further dried for 6 h at 40°C in a hot air oven (laboratory oven TT-9083; Techmel & Techmel).

Evaluation of polymeric microspheres

Scanning electron microscope (SEM) was used to determine the size (diameter) and shape of the polymeric microspheres.[34] Briefly, microspheres were applied to double-sided carbonated adhesive stills affixed to SEM stubs, and images were taken at 1 kV with ×5000 magnification in a SEM (Zeiss Ultra Plus; Carl Zeiss AG, Jena, Germany). The swelling index of the microspheres was also determined.

Ibuprofen-loaded microspheres (50 mg) were crushed with a mortar and pestle, and then suspended in a 10 mL of phosphate buffer with a pH of 7.2 and filtered after 24 h. The phosphate buffer was then used to dilute the filtrate before it was examined at 221 nm with a spectrophotometer. The following formula was used to compute the drug entrapment efficiency (Equation 8):

$$\text{entrapment (\%)} = \frac{\text{actual drug content}}{\text{theoretical drug content}} \times 100 \quad (8)$$

The paddle method was used in the in vitro dissolution tests with rotating at 100 rpm in 900 mL of 7.2 phosphate buffer at 37 ±0.5°C; 200 mg of ibuprofen was used as a model. Then, at regular intervals, 5-mL samples were removed and replaced with an equivalent volume of the new phosphate buffer. After the samples were diluted, the amount of ibuprofen released at 221 nm was measured using a ultraviolet-visible (UV/VIS) spectrophotometer (Spectrumlab 752s UV-VIS spectrophotometer; Wincom Company Ltd., Shanghai, China). To determine the mechanism of drug release, the dissolution data (i.e., the first 60% of drug release data) were fitted to the Korsmeyer–Peppas equation with DD Solver (Microsoft Excel 2016; Microsoft Corp., Redmond, USA).[35,36]

Results and discussion

Characterization of *Talinum triangulare* mucilage and pectin

The pectin of *T. triangulare* had a DE of 7.14% w/w, which suggests that *T. triangulare* is a low-methoxyl weight pectin. While LMP does not require a lot of sugar or acidity (low pH) to gel, it does require the presence of divalent cations.[29]

The elemental composition of the mucilage and pectin of *T. triangulare* is presented in Table 1. The highest elemental content in mucilage was sodium, whereas it was potassium in pectin. Copper, cadmium and lead were present in the polymers in minute, permissible quantities. According to The World Health Organization/The Food and Agriculture Organization of the United Nations (WHO/FAO), the permissible limits for copper, cadmium and lead are 40 mg/kg, 0.2 mg/kg and 0.3 mg/kg, respectively.[37] The presence of heavy metals has been previously reported in this plant.[38]

Table 1. Elemental properties of *Talinum triangulare* mucilage and pectin

Elements	Mucilage [mg/g]	Pectin [mg/g]
Ca	3.5760	7.8800
Fe	0.5332	0.8368
Na	20.3082	50.7970
Mg	17.5200	31.0000
K	10.8234	56.1130
Cd	0.0002	0.0017
Cu	0.0267	0.0823
Cr	0.0097	0.0658
Ni	0.0076	0.0309
Pb	0.0077	0.0623

The FTIR spectra shown in Fig. 1 indicate that the spectra for sodium alginate, pectin and mucilage of *T. triangulare* were distinct from that of ibuprofen, which had characteristic peaks at 1708–1729 cm^{-1} and 2955 cm^{-1}. The polymers showed no interaction with ibuprofen, even at the different ratios used, as indicated by the distinct fingerprint region (1500–500 cm^{-1}) of ibuprofen showing the aromatic ring and isobutyl moiety.[39] However, this was not the case with sodium alginate – the fingerprint region of ibuprofen was not clearly defined.

The mucilage particles were ovoid while pectin particles were irregularly shaped (Fig. 2) with *T. triangulare* mucilage having a smaller mean particle size than pectin (Table 2). Particles with smaller sizes and irregular shapes have better cohesiveness compared to oval and spherical particles; additionally, large particle sizes reduce cohesiveness and prevent particle packing.[40]

Packing behavior of a powder during various unit operations is described by its bulk density. The particle, bulk and tapped densities of pectin exceeded those of mucilage, according to the results shown in Table 2. This indicates that greater packing would be achieved with pectin than with mucilage, which would be important where shipping and packaging or low volume dosage forms are required.

The Hausner ratio and Carr's index are used to determine the flowability and compressibility of powders. A lower Carr's index indicates better flow and less compressibility,

Table 2. Material properties of *Talinum triangulare* mucilage and pectin

Polymer nature	Particle size [μm]	Particle density [g/cm³]	Bulk density [g/cm³]	Tapped density [g/cm³]	Hausner ratio	Carr's index [%]	Angle of repose (°)	Swelling power [%]
Mucilage	85.0 ±33.1	1.288	0.705	0.874 ±0.012	1.239	19.28	49.7	1.16
Pectin	197.0 ±78.3	1.569	0.711	0.882 ±0.014	1.240	19.38	48.0	1.09

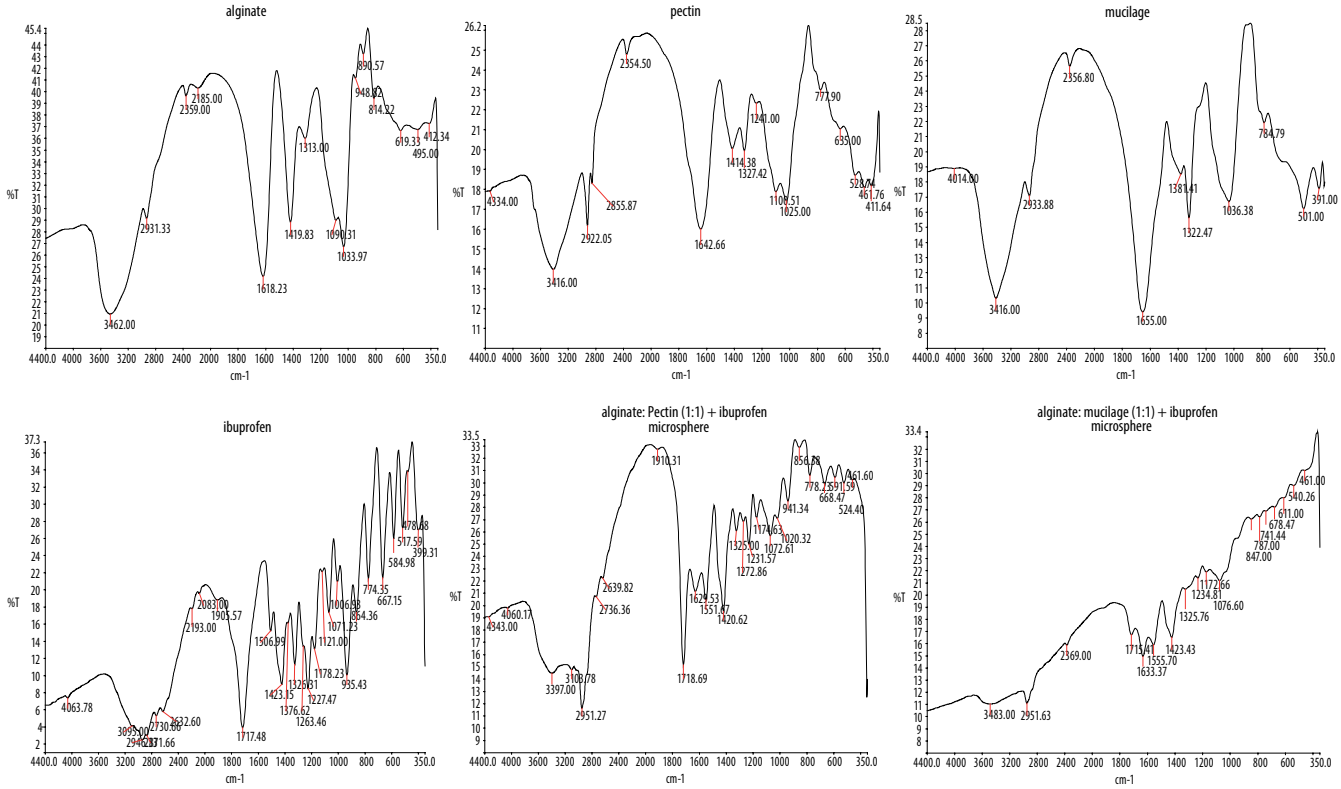


Fig. 1. Fourier-transform infrared spectroscopy (FTIR) spectra of polymers, ibuprofen and microspheres

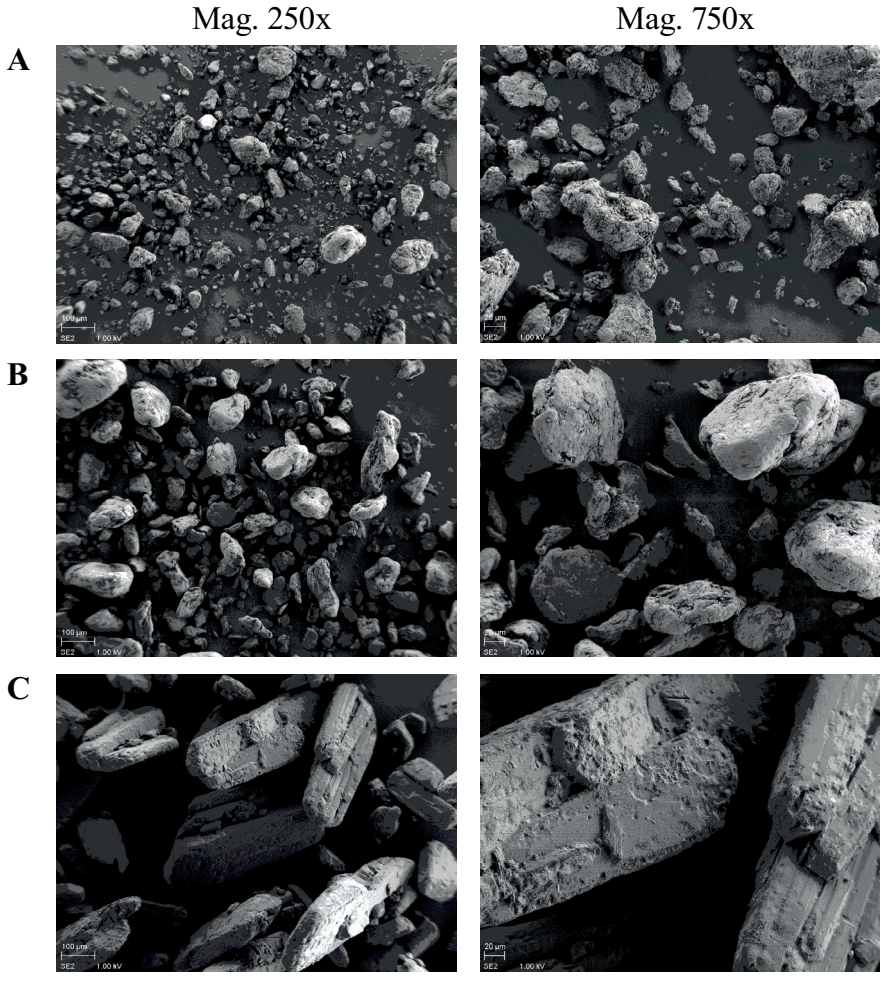


Fig. 2. Scanning electron microscope (SEM) images of (A) mucilage, (B) pectin polymers and (C) ibuprofen

while a higher Carr's index indicates less flow but better compression, implying a greater cohesiveness.[31] The Hausner ratio is related to the inter-particle friction: values greater than 1.25 signify that the material flow is passable, those greater than 1.35 indicate poor flow, while those greater than 1.5 indicate cohesiveness.[41] Our results show that *T. triangulare* mucilage has a better flowability than pectin, possibly due to the shape of the mucilage particles compared with the irregularly shaped pectin particles.

The angle of repose is used to measure the inter-particle force as well as the cohesiveness of materials. The rougher and more irregular the surface of the particles, the higher the angle of repose will be.[42] The angle of repose obtained for mucilage was higher than that of pectin, although pectin showed lower flow than mucilage.

Swelling power indicates the ability of a substance to hold fluid and its absorption behavior. It has generally been used to demonstrate differences between various types of materials.[43] The swelling power of mucilage was higher than that of pectin (Table 2).

Viscosity is the measure of fluid resistance to flow and a measure of the gradual deformation to shear or tensile stress.[44] The polymer blends exhibited non-Newtonian behavior in that their viscosity decreased with increasing shear rate for 1:1 blends and increased with increasing shear rate for 1:2 blends (Table 3). An increase in the concentration of either mucilage or pectin, with or without drugs, led to a decrease in the viscosity of the blends. Polymer blends with drug-containing pectin had higher viscosity than those containing mucilage at both ratios. There was no difference in the viscosity of the polymer blends at a ratio of 1:2 without the drug. Microspheres containing sodium alginate alone with or without drugs had the highest viscosity.

Properties of ibuprofen microspheres

The photomicrographs of the microspheres showed spherical to ovoid shapes at different ratios, with the microspheres with the ratio of 1:1 being more spherical (Fig. 3). This could be due to the decreased viscosity displayed at increased polymer concentrations, as compared to the viscosity of alginate. Therefore, increasing the concentration of the mucilage or pectin decreased the capacity of the polymer blend to form spheres. The surface morphology of the microspheres shown

Table 3. Viscosity analysis of polymeric blends

Polymer ratio [%]			Ibuprofen [%]	Viscosity [cP]
Sodium alginate	mucilage	pectin		50 rpm
100	–	–	–	19.28 ±7.30
50	50	–	–	1.98 ±2.30
50	–	50	–	1.96 ±4.00
34	66	–	–	0.62 ±2.30
34	–	66	–	0.62 ±2.30
33	33	–	34	1.70 ±2.80
33	–	33	34	2.40 ±0.00
22	44	–	34	0.42 ±2.80
22	–	44	34	0.74 ±2.80
66	–	–	34	24.85 ±8.30

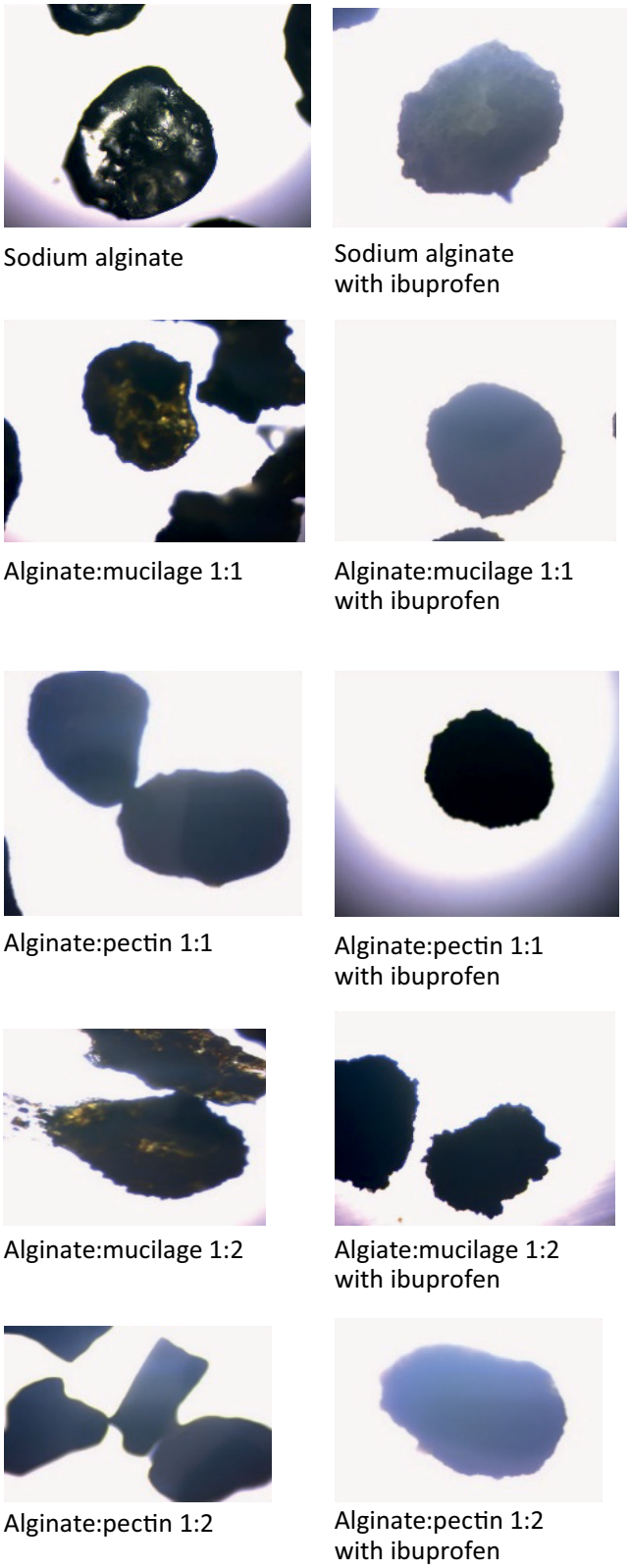
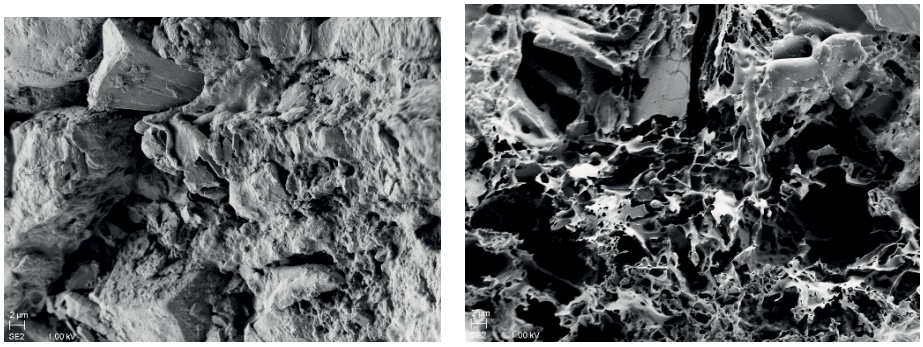
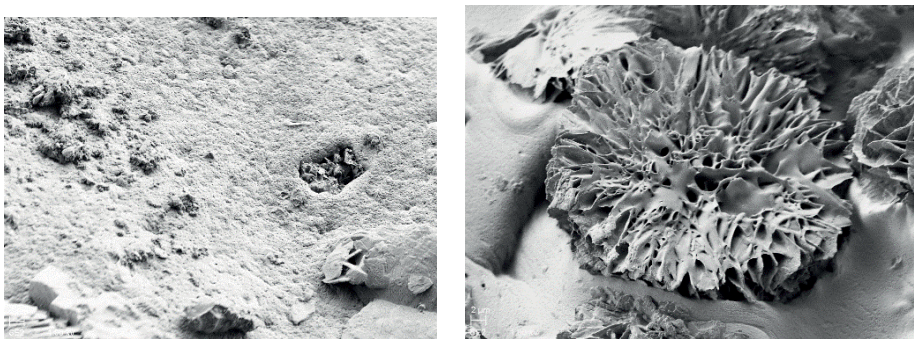


Fig. 3. Photomicrograph of all microspheres (×40 magnification)



Alginate:mucilage 1:1

Alginate:mucilage 1:1 with ibuprofen



Alginate:pectin 1:1

Alginat:pectin 1:1 with ibuprofen

Fig. 4. Scanning electron microscope (SEM) images of microsphere surfaces of polymer ratios 1:1 without and with drug (×5000 magnification)

in Fig. 4 indicates that the microspheres generally had rough surfaces, which is in agreement with previous studies.[33,45] Microspheres with polymer ratio of 1:1 showed smoother surface morphology. However, upon closer inspection with SEM, the surface revealed a spongy-like mesh for microspheres containing ibuprofen, while those without ibuprofen remained largely smooth. The microsphere sizes ranged within 929.4–1479.9 μm for mucilage and within 952.4–1652.6 μm for pectin (Table 4). In summary, the pectin-containing microspheres were generally larger.

Swelling power is described as the ability of the polymer matrix to absorb fluid and form a protective matrix. The swelling power (Table 4) of the microspheres was significantly higher ($p < 0.001$) in phosphate buffer at pH of 7.2 than in distilled water (pH 5.8). This indicated a greater absorption of fluid into the microspheres in the alkaline medium than in acidic medium, which suggests a greater effectiveness of the microspheres in the duodenum than in the stomach.[46]

Entrapment efficiency

Entrapment efficiency is the amount of drug entrapped or encapsulated within a matrix; it is an important parameter that describes the ability of the polymer blend or matrix to trap or hold drugs within it. Microspheres containing alginate:mucilage/pectin at a ratio of 1:1 had a greater entrapment of ibuprofen compared to a ratio

Table 4. Sizes and swelling index of all microspheres

Polymer ratio [%]			Microsphere size [μm]	Swelling [%]	
Sodium alginate	mucilage	pectin		phosphate buffer	distilled water
100	–	–	1311.9 ±120.3	349.5 ±91.2	2.0 ±0.0
50	50	–	1136.7 ±161.1	396.0 ±73.5	20.0 ±1.4
50	–	50	1276.4 ±269.2	71.0 ±65.0	27.0 ±5.65
34	66	–	1246.5 ±228.4	323.5 ±61.5	35.5 ±3.5
34	–	66	1083.8 ±131.4	51.0 ±0.0	2.5 ±0.7
33	33	–	1208.8 ±161.7	316.0 ±2.8	50.5 ±0.7
33	–	33	1090.4 ±115.4	284.5 ±43.1	24.0 ±2.8
22	44	–	1153.7 ±224.3	263.5 ±21.9	70.5 ±0.7
22	–	44	1400.9 ±251.7	142.0 ±26.8	22.0 ±1.4
66	–	–	1518.5 ±122.7	207.6 ±42.6	6.5 ±2.5

Table 5. Entrapment efficiency and dissolution time of ibuprofen in microspheres

Polymer ratio [%]			Shape of microsphere	Yield [%]	Entrapment efficiency [%]	Dissolution time [min]	
Sodium alginate	mucilage	pectin				t_{50}	t_{80}
50	50	–	spherical	77.37	55.71	45.0	98.0
50	–	50	spherical	99.67	60.43	66.0	–
34	66	–	irregular	69.10	45.94	32.0	66.0
34	–	66	oblong	88.63	39.57	31.0	37.8
100	–	–	spherical	97.93	48.64	68.0	100.0

of 1:2 (Table 5), thus indicating that an increase in the concentration of either mucilage or pectin did not enhance the entrapment of the drug. The entrapment efficiency of the alginate:mucilage/pectin ratio 1:1 was also higher than the alginate microspheres alone. Overall, the microspheres had an entrapment efficiency that ranged from 39.57% to 60.43%.

Release studies

The dissolution profiles of ibuprofen microspheres shown in Fig. 5 indicated different release properties based on the concentration of the pectin and mucilage present. The microspheres with a polymer blend of 1:1 had a longer release, whereas microspheres with a polymer blend of 1:2 had an immediate release, with the alginate:pectin blend of 1:2 having a faster release than the alginate:mucilage blend of 1:2. Microspheres containing a ratio of 1:1 of alginate:pectin had the slowest release rate, probably due to its higher viscosity, and a sustained release of over 2 h. All of the ibuprofen-loaded microspheres had t_{50} within the range of 31–68 min and t_{80} within the range of 37–100 min (Table 5), except for microspheres containing alginate:pectin at a ratio of 1:1, which failed to attain 80% drug release at 120 min. Fitting the microsphere dissolution data to the Korsmeyer–Peppas equation yielded correlation coefficients $R^2 \geq 0.977$ and $n > 0.89$. The drug release mechanism for all of the microspheres, irrespective of polymer or ratio, was the super case II transport (relaxation) mechanism.[47,48]

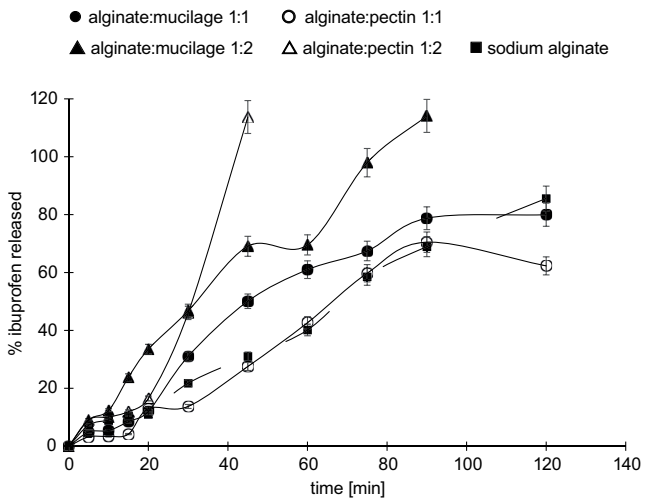


Fig. 5. Plots of the percentage of ibuprofen released against time [min] for alginate

Conclusions

The *T. triangulare* pectin particles displayed larger particle size and greater packing behavior than the mucilage, and were found to be low in methoxyl pectin. Interestingly, the mucilage particles showed better flow and swellability. The FTIR spectra showed no interaction of ibuprofen with the test polymers. Ibuprofen-loaded microspheres had significantly greater swelling in an alkaline medium. Polymer blends of pectin with ibuprofen had higher viscosity, and at a ratio of 1:1 had the slowest release of ibuprofen. Ibuprofen-loaded microspheres with polymer blends of 1:1 had a longer release of ibuprofen, whereas microspheres with polymer blends of 1:2 had immediate release of ibuprofen, even though they were all transported by the super case II transport mechanism. Therefore, the polymers of *T. triangulare* have use as matrices in microspheres depending on the type of drug release required.

ORCID iDs

Olufunke Dorothy Akin-Ajani https://orcid.org/0000-0002-8790-3653
Temiloluwa Mary Hassan https://orcid.org/0000-0003-2550-9615
Oluwatoyin Adepeju Odeku https://orcid.org/0000-0002-0732-1304

References

1. Reddy M, Manjunath K. Pharmaceutical applications of natural gums, mucilages and pectins: A review. *Int J Pharm Chem Sci.* 2013;2(3): 1233–1239. https://www.researchgate.net/publication/267329407. Accessed April 30, 2022.
2. Azad AKM. Isolation and characterization of pectin extracted from lemon pomace during ripening. *J Food Nutr Sci.* 2014;2(2):30. doi:10.11648/j.jfns.20140202.12
3. Paynel F, Pavlov A, Ancelin G, et al. Polysaccharide hydrolases are released with mucilages after water hydration of flax seeds. *Plant Physiol Biochem.* 2013;62:54–62. doi:10.1016/j.plaphy.2012.10.009
4. Levigne S, Ralet MC, Thibault JF. Characterisation of pectins extracted from fresh sugar beet under different conditions using an experimental design. *Carbohydr Polym.* 2002;49(2):145–153. doi:10.1016/S0144-8617(01)00314-9
5. Mollea C, Chiampo F, Conti R. Extraction and characterization of pectins from cocoa husks: A preliminary study. *Food Chem.* 2007;107(3): 1353–1356. doi:10.1016/j.foodchem.2007.09.006

6. Ciurzyńska A, Lenart A, Karwosińska J. Effect of quantity of low-methoxyl pectin on physical properties of freeze-dried strawberry jellies. *Pol J Food Nutr Sci*. 2015;65(4):233–241. doi:10.2478/pjfns-2013-0020
7. Yang JS, Mu TH, Ma MM. Extraction, structure, and emulsifying properties of pectin from potato pulp. *Food Chem*. 2018;244:197–205. doi:10.1016/j.foodchem.2017.10.059
8. Akin-Ajani O, Okunlola A. Pharmaceutical applications of pectin. In: Masuelli MA, ed. *Pectins: The New-Old Polysaccharides*. London, UK: IntechOpen; 2022. ISBN:978-1-83969-597-1.
9. Mellinas C, Ramos M, Jiménez A, Garrigós MC. Recent trends in the use of pectin from agro-waste residues as a natural-based biopolymer for food packaging applications. *Materials (Basel)*. 2020;13(3):673. doi:10.3390/ma13030673
10. Giacomazza D, Bulone D, San Biagio PL, Marino R, Lapasin R. The role of sucrose concentration in self-assembly kinetics of high methoxyl pectin. *Int J Biol Macromol*. 2018;112:1183–1190. doi:10.1016/j.ijbiomac.2018.02.103
11. Wu C, Pan LL, Niu W, et al. Modulation of gut microbiota by low methoxyl pectin attenuates type 1 diabetes in non-obese diabetic mice. *Front Immunol*. 2019;10:1733. doi:10.3389/fimmu.2019.01733
12. Alozie YE, Ene-Obong HN. Recipe standardization, nutrient composition and sensory evaluation of waterleaf (*Talinum triangulare*) and wild spinach (*Gnetum africanum*) soup "afang" commonly consumed in south Nigeria. *Food Chem*. 2018;238:65–72. doi:10.1016/j.foodchem.2016.12.071
13. Fontem D, Schippers R. Talinum triangulare (Jacq.) Willd: Record from PROTAbase. 2004. https://www.prota4u.org/database/protav8.asp?g=pe&p=Talinum+triangulare+(Jacq.)+Willd. Accessed May 27, 2022.
14. Enete A, Okon U. Economics of waterleaf (*Talinum triangulare*) production in Akwa Ibom State, Nigeria. *Field Actions Sci Rep*. 2010;4:1–12. https://journals.openedition.org/factsreports/438?file=1. Accessed April 30, 2022.
15. Adetuyi F, Dada I. Nutritional, phytoconstituent and antioxidant potential of mucilage extract of Okra (*Abelmoschus esculentus*), water leaf (*Talinum triangulare*) and Jews mallow (*Corchorus olitorius*). *Int Food Res J*. 2014;21(6):2345–2353. http://www.ifrj.upm.edu.my/21%20(06)%202014/41%20IFRJ%2021%20(06)%202014%20Adetuyi%20128.pdf. Accessed April 30, 2022.
16. Nya EJ, Okorie NU, Eka MJ. An economic analysis of *Talinum triangulare* (Jacq.) production/farming in southern Nigeria. *Trends Agricultur Econ*. 2010;3(2):79–93. doi:10.3923/tae.2010.79.93
17. Oluwalana I, Ayo J, Idowu M, Malomo S. Effect of drying methods on the physicochemical properties of waterleaf (*Talinum triangulare*). *Int J Biol Chem Sci*. 2011;5(3):880–889. doi:10.4314/ijbcs.v5i3.72167
18. Ikewuchi CC, Ikewuchi JC, Ifeanacho MO. Bioactive phytochemicals in an aqueous extract of the leaves of *Talinum triangulare*. *Food Sci Nutr*. 2017;5(3):696–701. doi:10.1002/fsn3.449
19. Kelechi A, Dorothy T. Economic study of tropical leafy vegetables in South-East of Nigeria: The case of rural women farmers. *Am J Agric Sci*. 2015;2(2):34–41. http://article.aascit.org/file/pdf/8920737.pdf. Accessed May 18, 2022.
20. Liao DY, Chai YC, Wang SH, Chen CW, Tsai MS. Antioxidant activities and contents of flavonoids and phenolic acids of *Talinum triangulare* extracts and their immunomodulatory effects. *J Food Drug Anal*. 2015;23(2):294–302. doi:10.1016/j.jfda.2014.07.010
21. Oliveira Amorim A, Campos de Oliveira M, de Azevedo Amorim T, Echevarria A. Antioxidant, iron chelating and tyrosinase inhibitory activities of extracts from *Talinum triangulare* leach stem. *Antioxidants*. 2013;2(3):90–99. doi:10.3390/antiox2030090
22. Oluwole O, Obote O, Elemo G, Ibekwe D, Adesioye T. Anti-inflammatory and anti-cancer properties of selected green leafy vegetables: A review. *Nutr Food Process*. 2021;4(8):1–5. https://www.auctoresonline.org/article/anti-inflammatory-and-anti-cancer-properties-of-selected-green-leafy-vegetables---a-review. Accessed May 18, 2022.
23. Swarna J, Lokeswari TS, Smita M, Ravindhran R. Characterisation and determination of in vitro antioxidant potential of betalains from *Talinum triangulare* (Jacq.) Willd. *Food Chem*. 2013;141(4):4382–4390. doi:10.1016/j.foodchem.2013.06.108
24. Biona K, Shen C, Ragasa C. Chemical constituents of *Talinum triangulare*. *Res J Pharm Biol Chem Sci*. 2015;6(1):167–171. https://www.researchgate.net/publication/268575472_Chemical_constituents_of_Talinum_triangulare. Accessed April 30, 2022.
25. Lara-Espinoza C, Carvajal-Millán E, Balandrán-Quintana R, López-Franco Y, Rascón-Chu A. Pectin and pectin-based composite materials: Beyond food texture. *Molecules*. 2018;23(4):942. doi:10.3390/molecules23040942
26. Andarwulan N, Faridah DN, Prabekti YS, et al. Dietary fiber content of waterleaf (*Talinum triangulare* (Jacq.) Willd) cultivated with organic and conventional fertilization in different seasons. *Am J Plant Sci*. 2015;6(2):334–343. doi:10.4236/ajps.2015.62038
27. Sanda M. Evaluation of quality and cholesterol level of eggs of laying hens placed on drinking water fortified with waterleaf (*Talinum triangulare*) mucilage. *Am Acad Sci Res J Eng Technol Sci*. 2015;13(1):81–87. https://core.ac.uk/download/pdf/235049679.pdf. Accessed April 30, 2022.
28. Ajala TO, Akin-Ajani OD, Ihuoma-Chidi C, Odeku OA. *Chrysophyllum albidum* mucilage as a binding agent in paracetamol tablet formulations. *J Pharm Investig*. 2016;46(6):565–573. doi:10.1007/s40005-016-0266-8
29. Okunlola A, Akindele O. Application of response surface methodology and central composite design for the optimization of metformin microsphere formulation using tangerine (*Citrus tangerina*) pectin as copolymer. *Br J Pharm Res*. 2016;11(3):1–14. doi:10.9734/BJPR/2016/25095
30. Akin-Ajani OD, Itiola OA, Odeku OA. Effect of acid modification on the material and compaction properties of fonio and sweet potato starches. *Starch–Stärke*. 2014;66(7–8):749–759. doi:10.1002/star.201300280
31. Carr R. Evaluating flow properties of solids. *Chem Eng*. 1965;18:163–168.
32. Ranganna S. *Handbook of Analysis and Quality Control for Fruits and Vegetable Products*. 2nd ed. New Delhi, India: Tata McGraw Hill; 2007. ISBN:978-0-07-451851-9.
33. Akin-Ajani O, Ikehin M, Ajala T. Date mucilage as co-polymer in metformin-loaded microbeads for controlled release. *J Excip Food Chem*. 2019;10(1):3–12. https://jefc.scholasticahq.com/article/8440-date-mucilage-as-co-polymer-in-metformin-loaded-microbeads-for-controlled-release. Accessed April 30, 2022.
34. Odeku OA, Aderogba AA, Ajala TO, Akin-Ajani OD, Okunlola A. Formulation of floating metronidazole microspheres using cassava starch (*Manihot esculenta*) as polymer. *J Pharm Investig*. 2017;47(5):445–451. doi:10.1007/s40005-017-0319-7
35. Ahmed L, Atif R, Eldeen T, Yahya I, Omara A, Eltayeb M. Study the using of nanoparticles as drug delivery system based on mathematical models for controlled release. *Int J Latest Tech Eng Manag*. 2019;8(5):52–56. https://www.ijltemas.in/DigitalLibrary/Vol.8Issue5/52-56.pdf. Accessed May 25, 2022.
36. Peppas NA, Sahlin JJ. A simple equation for the description of solute release. III. Coupling of diffusion and relaxation. *Int J Pharm*. 1989;57(2):169–172. doi:10.1016/0378-5173(89)90306-2
37. Elbagermi MA, Edwards HGM, Alajtal AI. Monitoring of heavy metal content in fruits and vegetables collected from production and market sites in the Misurata area of Libya. *ISRN Anal Chem*. 2012;2012(1–3):827645. doi:10.5402/2012/827645
38. Ukpabi C, Akubugwo E, Agbafor K, Wogu C, Chukwu H. Phytochemical and heavy metal composition of *Telfairia occidential* and *Talinium triangulare* grown in Aba, Nigeria, and environmental health implications. *Am J Biochem*. 2013;3(3):67–73. http://article.sapub.org/10.5923.j.ajb.20130303.01.html.Accessed April 30, 2022.
39. Dinte E, Bodoki E, Leucuta S, Iuga C. Compatibility studies between drugs and excipients in the preformulation phase of buccal mucoadhesive systems. *Farmacia*. 2013;61:703–712. https://farmaciajournal.com/wp-content/uploads/2013-04-art.09.dinte-703-712.pdf. Accessed April 30, 2022.
40. Okunlola A, Odeku OA. Effects of water yam and corn starches on the interacting variables influencing the disintegration of chloroquine phosphate tablets. *Dhaka Univ J Pharm Sci*. 2012;10(1):21–28. doi:10.3329/dujps.v10i1.10011
41. Staniforth J. Powder flow. In: Aulton ME, Cooper JW, eds. *Pharmaceutics: The Science of Dosage Form Design*. Edinburgh, UK-New York, USA: Churchill Livingstone; 1988. ISBN:978-0-443-03643-9.
42. Teixeira AZA. Compaction characteristics of the powder from the seed coat of tingui (*Magonia pubescens*). *Estud Biol*. 2007;29(68/69):277–282. doi:10.7213/reb.v29i68/69.22778

43. Kaur N, Garg T, Goyal AK, Rath G. Formulation, optimization and evaluation of curcumin-β-cyclodextrin-loaded sponge for effective drug delivery in thermal burns chemotherapy. *Drug Deliv*. 2016;23(7): 2245–2254. doi:10.3109/10717544.2014.963900
44. Ekolu S, Dundu M, Gao X. *Construction Materials and Structures: Proceedings of the First International Conference on Construction Materials and Structures*. Washington, USA: IOS Press; 2014. ISBN:978-1-61499-465-7.
45. Odeku OA, Okunlola A, Lamprecht A. Microbead design for sustained drug release using four natural gums. *Int J Biol Macromol*. 2013;58: 113–120. doi:10.1016/j.ijbiomac.2013.03.049
46. Sriamornsak P, Thirawong N, Korkerd K. Swelling, erosion and release behavior of alginate-based matrix tablets. *Eur J Pharm Biopharm*. 2007;66(3):435–450. doi:10.1016/j.ejpb.2006.12.003
47. Dash S, Murthy PN, Nath L, Chowdhury P. Kinetic modeling on drug release from controlled drug delivery systems. *Acta Pol Pharm*. 2010; 67(3):217–223. PMID:20524422.
48. Ghumman SA, Noreen S, Tul Muntaha S. *Linum usitatissimum* seed mucilage-alginate mucoadhesive microspheres of metformin HCl: Fabrication, characterization and evaluation. *Int J Biol Macromol*. 2020; 155:358–368. doi:10.1016/j.ijbiomac.2020.03.181