

# Synthesis Characterization and Optimization Study of Okra Gum Derivatives

<sup>1</sup>Savankumar H. Patel, <sup>2</sup>Yogesh M. Baj, <sup>3\*</sup>Nirmal K. Patel

<sup>1, 2, 3\*</sup>Department of Industrial Chemistry, Institute of Science & Technology for Advanced Studies & Research, Sardar Patel University, V. V. Nagar – 388120, Gujarat, (INDIA)

\*E-Mail: [drnirmalpatel@yahoo.com](mailto:drnirmalpatel@yahoo.com)

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**Abstract:** Okra is an associate of the mallow family which is related to cotton, hibiscus and hollyhock and it was extracted from the pods of hibiscus esculents using acetone as a solvent. The solution of okra gum in water has the highest viscosity among all the natural polysaccharide exposed till date. The extracted okra gum was further modified to okra phosphate which was further converted to hydroxyl propyl okra phosphate by using 3- chloro propionic acid. Reaction parameters such as reaction temperature, reaction time, volume of sodium hydroxide and amount of 3-Chloropropionic acid were optimized. Physical and chemical characteristics such as solubility, pH, moisture content and viscosity were observed for okra phosphate and hydroxylpropyl okra phosphate. Further extracted okra phosphate and of hydroxy propyl okra phosphate were characterized by Fourier transform infrared spectroscopy.

**Keywords:** Okra gum, Di-sodium hydrogen ortho phosphate, Acetone, 3-chloropropionic acid, Okra phosphate.

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## I. INTRODUCTION

Okra gum, obtained from the fruits of hibiscus esculentus, is a polysaccharide consisting of D-galactose, L-rhamnose and L-galacturonic acid, which is used as binder in pharmaceutical formulations which containing okra gum as binder showed a faster onset and higher amount of plastic deformation than those containing gelatin. By increasing binder concentration, the friability decrease, So that the crushing strength and disintegration times of tablets increased. The tablet produced from okra gum have higher crushing strength then gelatin produced tablets with longer disintegration times than those containing gelatin, so it was finally concluded from the results that okra gum may be a useful hydrophilic matrixing agent in sustained drug delivery devices [1], [2], [3].

Okra gum is modified to improve its application in control release drug delivery system. The derivatives of okra gum are prepared by reaction like alkylation, esterification, etc. The derivatives of okra gum are prepared under different experimental conditions such as time, temperature, etc. Different properties such as moisture regain, rate of hydration, solubility, viscosity and rheology of the derivatives have been studied. The properties depend upon polysaccharide chain length, the nature and degree of chemical modification [4], [5]. Chemical modification such as oxidation, acetylation, hydroxylpropylation and cross-linking provide efficient route not only to reduce the drawbacks but also to improve on the physicochemical properties and to introduce new properties for different applications [6].

Okra gum or its derivatives are used in pharmaceutical industries as gelling, viscosifying, suspension, stabilization, emulsification, preservation, water retention/water phase control, binding, clouding/bodying, process aid, pour control for suspensions and anti-acid formulations. However, okra gum derivatives are used for controlling the release of drugs in the gastrointestinal tract such as, carrier for colon targeted drugs for anticancer drugs and for oral rehydration solutions in the treatment of cholera in adults. Also, it has many applications in various industries such as paper, food, textile, cosmetic; nutrition and animal feed [7], [8], [9], [10].

Modified okra is used in the frozen food industry. Okra phosphate has its application in manufacturing of desserts, sweet creams, cakes, sauces, and yogurt and in non-food industry as additive [11], [12], [13].

Hydroxypropyl okra phosphate used in cosmetic and beauty products because of their ability to serve as emulsifier and surfactant. It impart smooth and velvety feel that's the reason it is used in hair products. Hydroxypropyl okra phosphate is popular ingredient in cosmetics because of its versatility which it stabilizes emulsion by giving instant viscosity and provides smooth, thick feeling. There is no adverse safety warning for hydroxypropyl okra phosphate [14], [15], [16].

In this present work, okra phosphate is synthesized from okra gum and hydroxypropyl okra phosphate is synthesized from okra phosphate. The synthesized okra phosphate and hydroxypropyl okra phosphate is characterized by moisture content, pH, solubility test, viscosity. Degree of substitution and effect of different parameters viz. time, temperature, effect of NaOH and effect of 3-chloropropionic acid on hydroxypropyl okra phosphate is studied.

## II. MATERIALS

Okra fruit was purchased from local vegetable market. Di-sodium hydrogen ortho phosphate, sodium hydroxide, 3-chloropropionic acid, acetone and methanol were purchased from Atul Chemical, Anand. Solvents and other laboratory chemicals were used after routine purification and they were of analytical reagent grade. Double distilled water was used.

## III. METHODS

### A. Extraction of Okra Gum:

500 g of unripe and tender okra fruit was washed and sliced with a knife. The sliced mass was soaked in distilled water overnight. After soaking, white muslin cloth was used to filter out the viscous gum extract. Acetone was added to precipitate the gum at a ratio of 1/3 parts of acetone to 1 part of the gum extract. The precipitated gum was dried in a desiccator containing anhydrous calcium chloride for approximately 15 days. Size reduction and screening of the dried gum were carried out using a stainless steel grinder and no. 30 stainless steel mesh sieve. Airtight powder bottles were used to store the undersize fractions.

### B. Purification of Okra Gum:

2 g of the okra gum was boiled with 8 mL of 70% (v/v) ethanol for 1 h with reflux. The sample was filtered, washing with 95% (v/v) ethanol and dried at 60 °C for 3 h in vacuum oven.

### C. Preparation of Okra Phosphate:

Take 10 g of okra gum and 3 g of di-sodium hydrogen okra phosphate into 3-neck flask. 10 ml of 3 M NaOH is added drop wise within 5 min with constant stirring for 1h. Then pH of the solution formed is made neutral by adding acid. Methanol is added into solution to precipitate out okra phosphate. It is then centrifuged and product is dried at room temperature for 12 h, or in oven at 40<sup>0</sup> to 45<sup>0</sup> C.

### D. Purification of Okra Phosphate:

Okra phosphate was purified with distill water. Product remained dialyzed as compared to distilled water for 48 h and then okra phosphate was precipitated with methanol. Finally product was dried in oven at temperature of 40-45°C.

### E. Moisture Content of Extracted Okra Phosphate and Hydroxypropyl Okra Phosphate:

The moisture content of the sample is calculated using the following equation:

$$\%W = \frac{A - B}{A} \times 100$$

Where:

%W = Percentage of moisture in the sample,

A = Weight of wet sample (g),

B = Weight of dry sample (g)

### F. pH Determination of Extracted Okra Phosphate and Hydroxypropyl Okra Phosphate:

1 % w/v dispersion of the sample in water was stirred consistently for 5 minutes and pH was determined using a pH meter with built in magnetic stirrer model equiptronics-614.

**G. Solubility Test of Extracted Okra Phosphate and Hydroxypropyl Okra Phosphate:**

Solubility of the extracted okra gum was evaluated qualitatively by stirring 10 mg of okra powder in 10 mL water, acetone, chloroform, dimethyl sulfoxide (DMSO), dimethyl formamide (DMF) and methanol. Solubility was determined by visual observation of the solute.

**H. Viscosity of Extracted Okra Phosphate and Hydroxypropyl Okra Phosphate:**

Viscosity of okra gum at 1 % and 0.5 % concentrations was measured using the Gardner – bubble viscometer.

**I. Preparation of Hydroxypropyl Okra Phosphate from Okra Phosphate:**

About 5 g of okra phosphate is dissolved in 100 ml water. The solution is taken into 3-neck flask. 15 ml of 2 M NaOH is added drop wise with constant stirring for 15 minutes. 4 ml of 3-chloro propionic acid is added drop wise. The mixture is then stirred for about 4h after the completion required amount of methanol is added into solution to precipitate out product. It is then filtered off and dried in oven at 45 °C.

**J. Purification of Hydroxy Propyl Okra Phosphate:**

Hydroxy propyl okra phosphate was purified with water. Product remained dialyzed as compared to distilled water for 48 h and then hydroxy propyl okra phosphate was precipitated with methanol. Finally product was dried in oven at temperature of 40-45 °C.

**K. Degree of Substitution of Hydroxyl Propyl Okra Phosphate:**

Hydroxy propyl okra phosphate is acidifying by ion exchange. The solution is then divided into two parts.

Solution 1: It was taken into previously weighted beaker. The solution was heated until all solvents on hot plate evaporated. Then solution is cooled and hydroxypropyl okra phosphate was weighed.

Solution 2: It was titrated against a standard solution of NaOH. The burette reading was noted down and degree of substitution find out by following equation.

$$\text{Degree of substitution} = 0.162 B / (1 - 0.58 B)$$

Where, B= Volume of NaOH/Weight of sample

This is the characterization data of hydroxy propyl okra phosphate.

**IV. RESULT AND DISCUSSION****L. Optimization study parameters of hydroxyl propyl okra phosphate****Effect of NaOH:****TABLE 1:**

Sr. NO	NaOH, (mL)	Temperature (°C)	3-Chloropropionic acid, (g)	Time (h)	Degree Of Substitution
1	1	45	4	4	1.19
2	2	45	4	4	2.10
3	3	45	4	4	3.18
4	4	45	4	4	3.18

Hydroxyl propyl okra phosphate was carried out at variable amount of NaOH from 1-4 mL (TABLE 1). But degree of substitution (DS) increased as a result of increasing the amount of NaOH up to 3 mL. There was no change in the degree of substitution when NaOH was used in the amounts of 3 mL and 4 mL. So we found 3 mL NaOH is the best proportion. Increase in amount of NaOH leads to the alkali degradation of polymer. The lower amount of NaOH leads to lower number of free hydroxyl group deprotonated to form alkoxide which was resulted into lower value of degree of substitution.

**Effect of Temperature:****TABLE 2:**

Sr. NO	Temp. (°C)	NaOH (ml)	3-Chloropropionic acid (g)	Time(h)	Degree Of Substitution
1	35	3	4	4	2.7
2	40	3	4	4	2.98
3	45	3	4	4	3.18
4	50	3	4	4	3.18

Hydroxyl propyl okra phosphate was carried out in the range of 35-50 °C, keeping other limits constant (TABLE 2). It can be perceived from the table that degree of substitution improved with increase in temperature from 35-50 °C. But the degree of substitution obtained at 50 °C was almost same as obtained at 45 °C. Beside maintaining temperature higher than 45 °C was likely to increase the production cost. So we selected 45 °C as the best reaction temperature.

**Effects of 3-chloropropionic acid:****TABLE 3:**

Sr. NO	3-Chloropropionic acid (g)	NaOH (ml)	Temp. (°C)	Time (h)	Degree Of Substitution
1	1	3	45	4	2.3
2	2	3	45	4	2.58
3	3	3	45	4	2.9
4	4	3	45	4	3.18
5	5	3	45	4	3.19

Hydroxyl propyl okra phosphate was carried out by varying amount of 3-Chloropropionic acid from 1-5 g (TABLE 3). But degree of substitution increase as a result of increasing the amount of 3-Chloropropionic acid up to 5 g. The reaction remains constant by further increase in the amount of 3-Chloropropionic acid there was no change in the degree of substitution with increase in 3-Chloropropionic acid, so we found 4 g 3-Chloropropionic acid is the best proportion. If the less amount of 3-Chloropropionic acid used then it leads to unneutralization of alkali so the optimum 3-Chloropropionic acid (4 g) was used to leads the favorable neutralization of alkali and to better formation of Hydroxyl propyl okra phosphate having higher degree of substitution value.

**Effects of Time:****TABLE 4:**

Sr. NO	Time(h)	NaOH(ml)	Temp. (°C)	3-chloropropionic acid (g)	Degree Of Substitution
1	1	3	45	4	2.1
2	2	3	45	4	2.5
3	3	3	45	4	2.82
4	4	3	45	4	3.18
5	5	3	45	4	3.24

Hydroxyl propyl okra phosphate was carried out in the duration of 1-5 h, keeping other limits constant (TABLE 4). It can be settled from the table that degree of substitution improved with increase in time from 1-5 h. But degree of substitution was almost same at the end of 4 h and 5 h. So we selected 4 h is the best reaction time. Besides, carrying out the reaction for 5 h (i.e. one h more) was likely to increase the production cost.

**Characterization study:**

Characterization study for the extracted Okra phosphate and extracted Hydroxypropyl okra Phosphate was carried out and the values are listed in the TABLE 5.

**M. Characterization study of Okra phosphate and Hydroxypropyl okra Phosphate:**

TABLE. 5

Properties	Extracted okra gum	Okra phosphate	Hydroxypropyl okra Phosphate
Moisture content	10.96%	9.3%	8.3%
pH*	6.59	9.10	9.12
Viscosity**	53.6 cSt (A tube) and 68.8 cSt (B tube)	92.7 cSt (A tube) and 102.9 cSt (B tube)	122.7 cSt (A tube) and 151.9 cSt (B tube)
Solubility	Soluble in water. Insoluble in acetone, chloroform, methanol, DMSO and DMF.	Soluble in water and DMSO. Insoluble in Chloroform, Methanol, Acetone, DMF	Soluble in distill water and isopropyl alcohol. Insoluble in Methanol, DMSO, DMF, Chloroform

\*For 1% w/v solution, \*\* 1% aqueous solution used in tube A & 0.5% in tube B (Gardner bubble viscometer method)

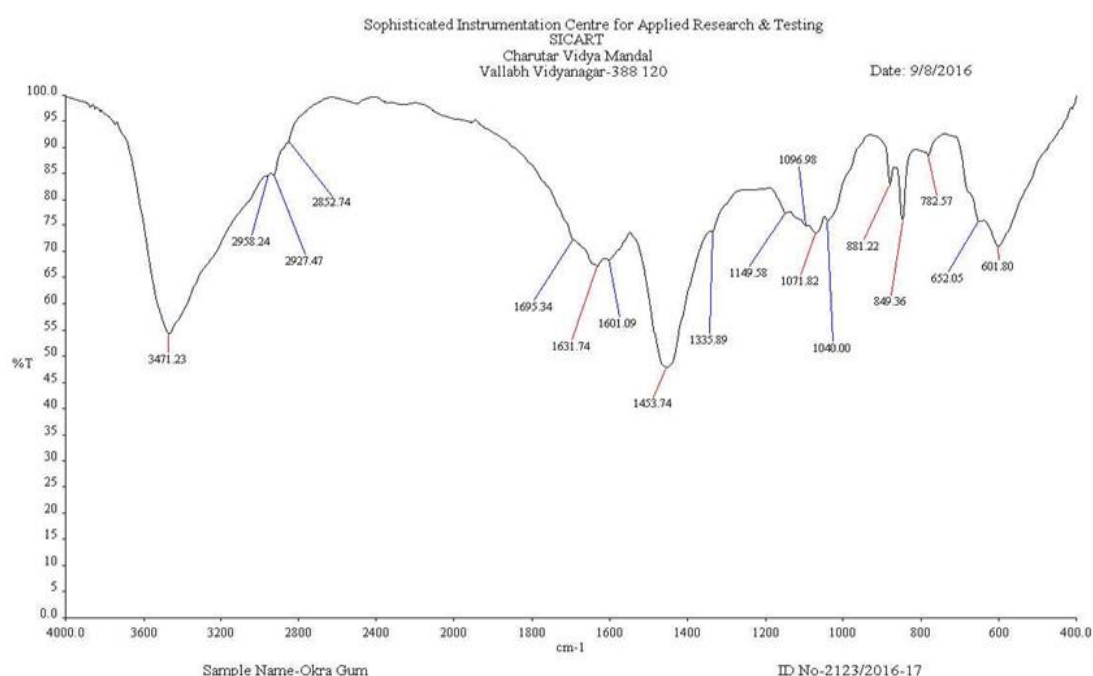
**N. FTIR spectrums of okra gum:**

Fig. 1

FTIR spectrums of okra gum were shown in Fig. 1. The absorption band at  $3471.23\text{ cm}^{-1}$  in IR spectrum of okra gum showed intensity for the hydroxyl group present in okra gum. The sharp absorption band located at  $2927.47\text{ cm}^{-1}$  may be attributed to CH group stretching.

**O. FTIR spectrums of okra phosphate:**

FTIR spectrum of okra phosphate gum is shown in Fig. 2. The IR spectrum of okra phosphate gum showed a reduced intensity of the absorption band located at  $3438.10\text{ cm}^{-1}$ , as compared to okra gum IR spectrum. This happened due to -OH stretching which indicated that some -OH group were phosphate. The sharp absorption band located at  $2925.07\text{ cm}^{-1}$  may be attributed to CH group stretching. The medium-weak absorption band located at  $1154.87\text{ cm}^{-1}$  may be attributed to P=O group stretching while, medium-weak absorption band located at  $763.00\text{ cm}^{-1}$  may be attributed to P-C group deformation band and stretching, which is absent in the okra gum spectra.

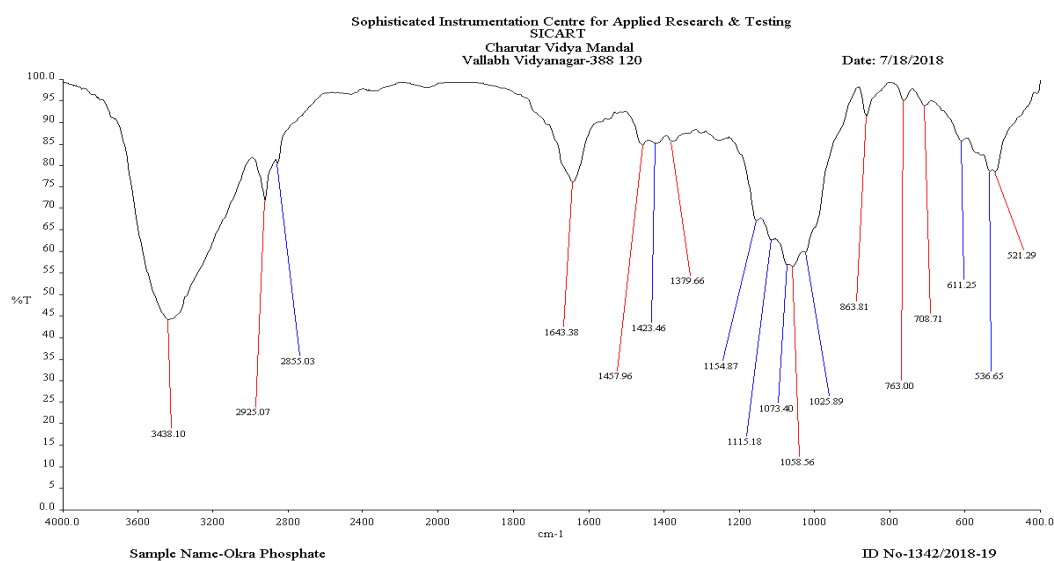


Fig. 2

#### P. FTIR spectrums of Hydroxypropyl okra Phosphate:

FTIR spectrums of hydroxypropyl okra phosphate were shown in Fig. 3. The absorption band at  $3429.00\text{ cm}^{-1}$  in IR spectrum showed intensity for the hydroxyl group present in hydroxypropyl okra phosphate. The sharp absorption band located at  $2925.36\text{ cm}^{-1}$  may be attributed to CH group stretching. The medium-weak absorption band located at  $1156.42\text{ cm}^{-1}$  may be attributed to P=O stretching while, medium-weak absorption band located at  $762.79\text{ cm}^{-1}$  may be attributed to P-C deformation band and stretching. The absorption band located at  $1455.80\text{ cm}^{-1}$  and  $1419.13\text{ cm}^{-1}$  may be attributed to  $-\text{CH}_2-$  stretching presence in propyl group while, absorption band attributed at  $1375.68\text{ cm}^{-1}$  confirms the  $-\text{CH}_3$  group presence in propyl group.

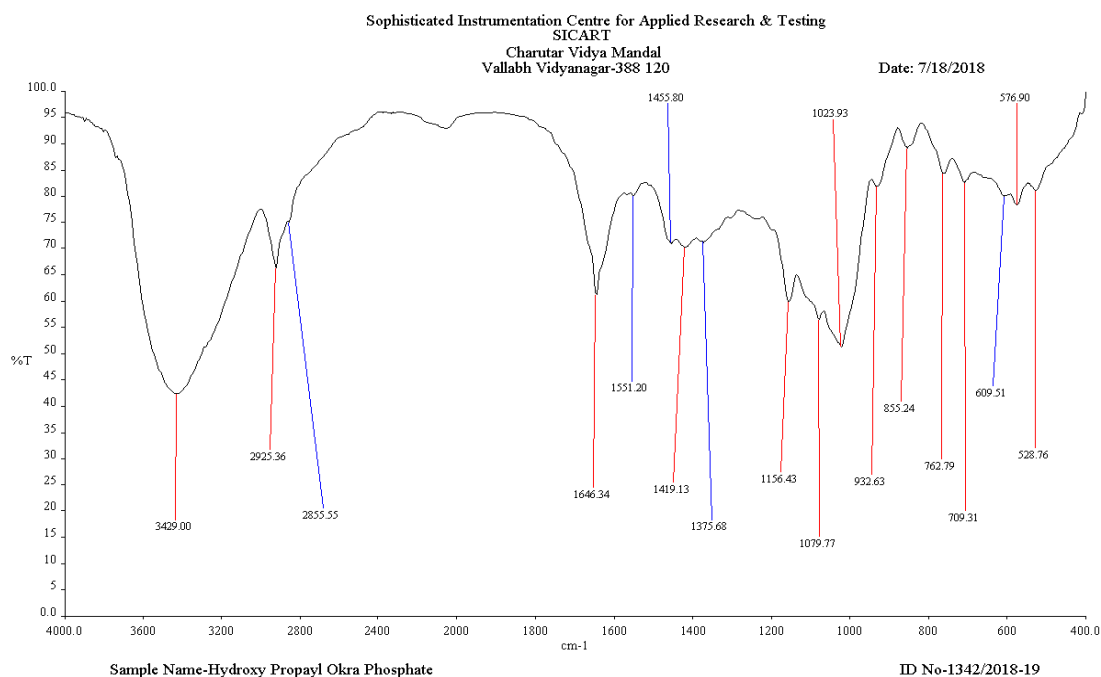


Fig. 3

## V. CONCLUSION

Okra gum was successfully extracted from okra pods by solid-liquid extraction methods, Fruitful extraction was carried out and the extracted compound was confirmed by FTIR Further okra phosphate and hydroxy propyl okra phosphate was prepared fruitfully from extracted okra gum using di-sodium hydrogen okra phosphate and 3-chloro propionic acid as reagent the structure was confirmed by FTIR. Reaction parameters such as temperature, time, volume and concentration of solvent were optimized. The optimum degree of substitution of 3.18 was obtained by carrying out reaction at 45°C for 4 h by addition of 3 ml of NaOH and 4 ml of 3- chloropropionic acid. Ph, moisture content, and solubility test of resulted product was successfully carried which was enhanced by the modification of okra, okra gum can also be modified to various pharmaceutical derivatives as drug carrier as it contain hydroxyl grope which can be easily replaced by any other functional grope carrying out various chemical reaction to increase their property's

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