RESEARCH PAPER

Formulation and Evaluation of Sustained Release Micro Beads of Gellan Gum and Apple Peel Pectin Composite Using Aceclofenac as a Model Drug

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ABSTRACT
This current communication describes the micro beads composed of apple peel pectin (APP) and gellan gum (GG) blends for sustained Aceclofenac (AC) release over a prolonged period. As a model drug candidate, AC was investigated. These AC-loaded GG-APP composite micro beads presented 46.27 □ 2.08 to 68.66 ± 2.74% entrapment efficiency and 702.84 □ 21.53 to 987.46 □ 37.57 µm average particle size. The GG-APP composite micro beads demonstrated a sustained-prolonged release over 8h.
Keywords: Aceclofenac; Apple peel pectin; Gellan gum; Micro beads; Sustained drug release
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INTRODUCTION

GG is well-known as anionic charged gum, which is aqueous [1, 2]. soluble. The deacetylated type of GG has been reported for outstanding ability for iono tropic gelation by divalent and trivalent metal ions like Ca^{2+} and Al^{3+}[4].This hydrogel-formation potential of de acetylated GG is being employed to design many sustained release matrices, even in drug delivery[5,6].Although sustained drug-releasing hydrogel beads made of GG have already been investigated over the last few years, the uses of bio polymeric blends containing GG and other biopolymers are currently being used to improve the requisite characteristics of drug delivery matrices like matrix stability, swelling, entrapment of drugs, releasing of drugs, and so on[1,7].Such kinds of hydrogels made of GG-other biopolymer blends has already been investigated to minimize the untimely releasing of drugs in intestinal pH with more specifically[6].

Apple peel pectin (APP) is a natural polysaccharide derived from the apple peels[8, 9]. It is reported as aqueous soluble polysaccharide. Over the past few years, a few research endeavors have been attempted to use APP as pharmaceutical excipients, including drug delivery dosage form designing[10-12].The goal of the present study was fabrication, characterization, and evaluation of the IG composite micro beads that release drugs with sustained action using de acetylated APP and GG. In the available literature, such IG GG-APP composite micro beads are not available, till date. In the present research, aceclofenac (AC) was used as a model drug, which is extensively used in pain and inflammation management, specifically inosteoarthritisas well as rheumatoid arthritis treatments[3, 13, 14,15,16,17,18]. The plasma half-life (elimination) of AC is about 4 hours, and the recommended daily dose is 200 mg[4].

MATERIALS AND METHODS

Materials:

The source of AC [$C_{16}H_{13}C_{l2}NO_4$] was Drakt Pharmaceutical Pvt. Ltd. in India. Absolute ethanol (Sigma Aldrich, India), citric acid (Merk, India), and de acetylated GG (SRL India Ltd., India) were employed. In this study, APP was extracted from apple peel material that was gathered in October 2019 from a local market in Jharpokharia, Mayurbhanj district, Odisha, India. The remaining compounds were all commercially accessible and of analytical quality.

Extraction of APP

The apples were washed several times to remove the dirt and other materials. They were chopped into small pieces and blanched in boiling water for 5 min. Following a 30-minute treatment in warm absolute ethanol to extract oil from the peel material, the produced material was filtered by hand using muslin cloths, and then it was cleaned. To get rid of extra water, the cleaned material was compressed with manual pressure. The resulting material was allowed to dry in the shade for a few days and then converted into fine powder after complete drying. Weighed amount (5 g) of powdered peel was added to a 250 ml conical flask, 150 ml of citric acid was admixed

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while the pH level remained at 2. After that, the mixture was heated for one hour at 80°C. Muslin cloths were used to filter the hot acid extract. The filtrate was then allowed to cool at room temperature. Then, at 4°C for three hours, an equal volume (1:1) of 99.1% ethanol was used to coagulate the pectin-containing aqueous extract. The resulting precipitate, or ethanol-insoluble fraction, was extracted by filtration and centrifugation. The resulting substance was first cleaned with 55% ethanol and then with 75% ethanol. A tray drier set to 40 ± 1°C was used to dry the ethanol-washed material. The dried APP was kept in desiccators for further study.

Preparation of micro beads enclosing AC

Micro beads of GG-APP containing AC were prepared by Al^{3+}-ion induced ionotropic gelation process[3,4]. Briefly, by heating at 60 ± 0.5°C with continuous stirring, de acetylated GG solutions (aqueous) were prepared, while pectin aqueous solutions were prepared using extracted APP by heating at 40 ± 0.5°C with continuous stirring. The GG-APP solutions were prepared by thoroughly mixing the two polymeric aqueous solutions (GG and APP) with stirring. To prepare the GG-APP solution-mixtures, 2% w/v concentration of both the polymers (namely GG and APP) were used in all these micro beads formulations. In each of these micro beads, AC (in the necessary amount) was added to the prepared GG-APP solution mixtures. A homogenizer (Remi Motors, India) was used to mechanically homogenize the final mixtures of AC-containing GG-APP solutions for thirty minutes at 500 rpm. Using an 18-G needle, the resulting polymer-AC dispersions were introduced drop by drop into aqueous solutions of AlCl3 (at the necessary concentration) for 15 minutes. After decanting the resultant wet hydrogel beads. It dry overnight at 40 ± 1°C in a tray dryer. For future research, the dried GG-APP beads were kept in desiccators.

Measurement of DEE

Samples of prepared GG-APP micro beads (GG-APPM) enclosing AC were sampled and pulverized into a powder using clean mortar and pestle. Powdered 10 mg micro bead samples were mixed to a clean glass beaker for 24 hours at 37°C ± 0.5°C. Following an overnight interval, the mixture was continuously stirred for 30 minutes

Exact quantity of AC in GG-APPM

$$\text{DEE (\%)} = \frac{\text{Exact quantity of AC in GG-APPM}}{\text{Theoretical content of AC in GG-APPM}} \times 100$$

Measurement of particle size

An optical microscope (Olympus) was used to measure the particle size of 100 particles for dried GG-APPM containing AC. A stage micrometer was used to calibrate the micrometer [4]. The data is presented as mean ± SEM.

Scanning -Electron -Microscopy (SEM):

Dried GG-APPM samples were gold coated and examined using Scanning Electron Microscope

Fourier Transform-infrared (FTIR) Spectroscopy :

A clean mortar and pestle was used to powder the dried GG-APPM samples.

***In Vitro* evaluation study:**

The release of AC from Al3+-ion induced IG GG-APPM was evaluated at 37°C ± 0.5°C at 50 rpm [3,4]. The in vitro drug release study was conducted for two hours in 0.1 N HCl (pH 1.2) and then for six hours in an alkaline dissolution medium (in this case, phosphate buffer, pH 7.4). Five milliliter aliquots were taken out at regular intervals, and the dissolution equipment was immediately replaced with an equivalent amount of fresh medium.

Statistical analysis

Data obtained in this research were analyzed by Med Calc software.

RESULTS AND DISCUSSION

DEE

The estimated DEE (%) of AC-containing GG-APP Mranged 46.27 ± 2.08 to 68.66 ± 2.74% (**Table 1**). From the obtained results, it was evident that the polymer contents (contents of GG and APP) and $AlCl_3$ (anionic cross-linker) influenced DEE (%). DEE (%) values were found to be increased with the increasing polymer contents and declining cross-linker concentrations.

Particle size

The AC-containing GG-APPM was ranged702.84 ± 21.53to 987.46 ± 37.57 µm (**Table 1**)., GG and APP) and declining $AlCl_3$(anionic cross-linker) concentrations employed for AC-containing GG-APPM preparation. Increase in mean particle size with increasing polymer contents by the fact of hydrodynamic viscosity. In addition, decline in micro beads sizes was noticed with the increase in concentration of $AlCl_3$ solutions (aqueous). This may be explained by the fact that more concentrated anionic cross-linker ions improve the degree of cross-linking in IG bio polymeric-gel matrices.

SEM Analyses

The SEM photograph of AC-containing GG-APPMis shown in **Fig. 1**.It indicated the irregular shape of prepared microbeads.The prepared GG-APPM with AC showed extremely rough surface morphological characteristics.

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***In vitro* drug release**

AC-containing GG-APPM showed a prolonged pattern of AC released over a period of 8 h (**Fig. 3**). The findings showed that *in vitro* AC release from GG-APPM was comparatively slower in acidic pH (1.2) for the first two hours and comparatively faster in alkaline pH (7.4). GG-APPM containing AC may bind to water well, resulting in a viscous gel-like structure on its surface. In order, to demonstrate the continuous release of AC (*in vitro*), this viscous gel-structure development may have formed a barrier on the pores on the surface of the micro beads. As the amount of polymers (GG and APP) and the con^{c} of $AlCl_3$ solutions (aqueous) increased.

Release Kinetics and Mechanism

Several mathematical models, were used to kinetically analyze the *in vitro* data of AC-containing GG-APPM [13–16]. In these model equations, The aforementioned models are k_0, k_1, k_h, and $K_{k\text{-}p}$, respectively. "n" stands for the release exponent. For spherical matrices, the Fickian mechanism is indicated by $n \leq 0.43$. The case-II transport mechanism is indicated by $n \geq 0.85$, whereas the non-Fickian mechanism is indicated by $n = 0.43–0.85$.[17-20].

Table 2 displays the outcome of the curve fitting. The zero-order model was followed by the *in vitro* AC release from A-2, A-4, A-5, A-6, A-8, and A-O GG-APPM containing AC ($R^2 = 0.9804$ to 0.9946). In the case of A-5 and A-6 GG-APPM containing AC, it was found that the *Korsmeyer. Peppas. Model (*$R^2 = 0.9970$ and 0.9865), *The first-order model* ($R^2 = 0.9809$ to 0.9919) was used for the other GG-APPM that contained AC (A-1, A-3, and F-7).The n values AC-containing GG-APPM (A-2, A-4, A-5 to A-6) . In contrast, A-1, A-3, A-7 and A-8 GG-APPM containing AC demonstrated *Non-Fickian releasing* mechanism indicating diffusion and swelling controlled releasing.

4 CONCLUSION

In the current research, Al^{3+}-ion cross linked IG composite micro beads composed of GG and APP blends was successfully prepared. These various AC-containing GG-APPM demonstrated 46.27 ± 2.08 to 68.66 ± 2.74% AC entrapment efficiency and 702.84 ± 21.53 to 987.46 ± 37.57 µm average particle size. The loaded AC from different AC-loaded GG-APP composite micro beads demonstrated a sustained release over 8 h. The sustained release potential of the prepared AC-containing GG-APPM could be beneficial in terms of decreased dosing intervals as well as patient compliances. Therefore, AC's IG GG-APPM can be considered for prolonged, sustained AC release. Other medications that need intestinal release may also benefit from the sustained delivery of these Al^{3+}-ion cross-linked GG-APPM.

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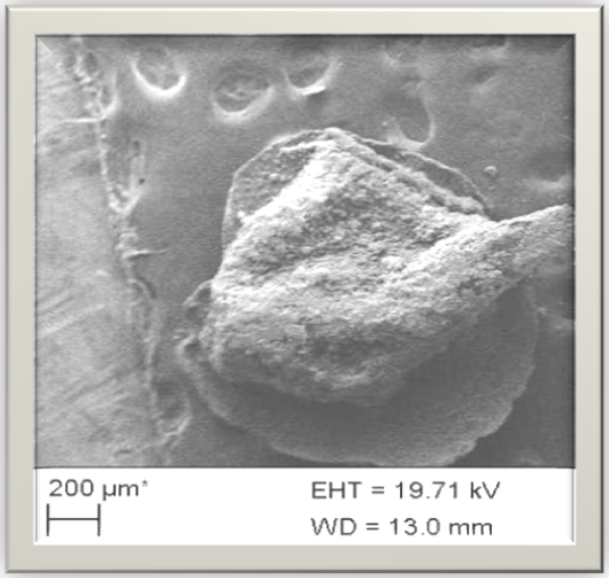

Fig 1. SEM photograph of Formulation A-O

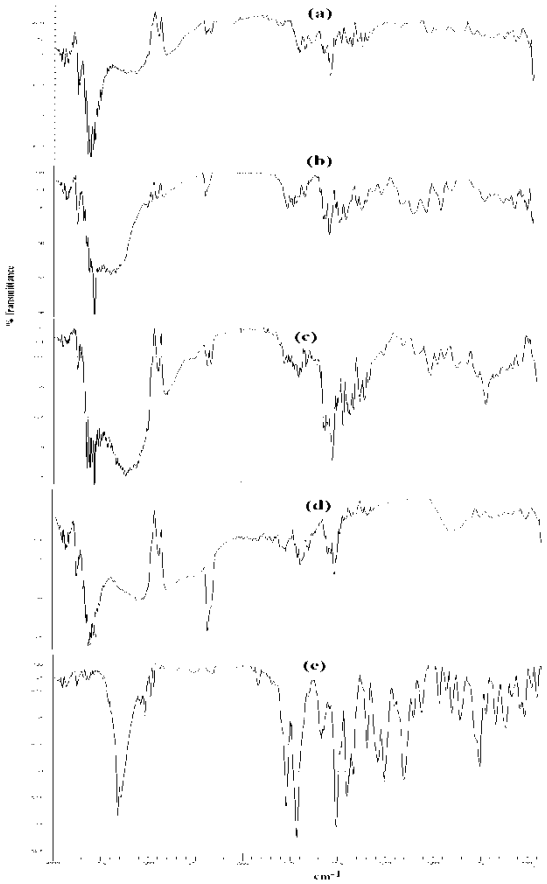

Fig 2. Spectral Analysis (FTIR) of different samples (a) GG (De -acetylated), (b) APP, (c) GG-APPM without AC loading, (d) AC-containing GG-APPM and (e) pure AC.

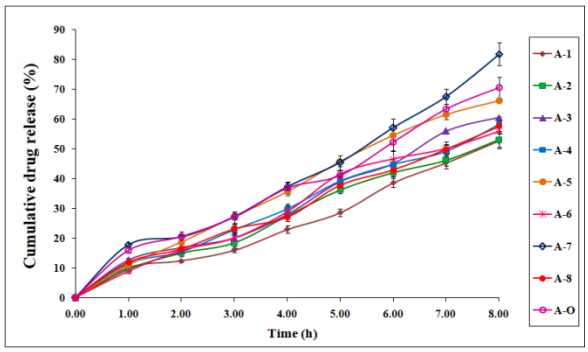

Fig 3. Release of AC-containing GG-APPM [Mean ± S.D.; n = 3)

Table 1. Formulation variables of different AC-containing GG-APPM with results of DEE (%) and mean particle size

Formulations	Parameters			DEE (%)*,#	Mean diameter (µm)**
	Amount (mg)		Concentration (%)		
	GG	APP	$AlCl_3$		
A-1	280	50	5	33.82 ± 1.27	824.47 ± 36.44
A-2	280	50	3	50.08 ± 2.22	898.54 ± 38.27
A-3	280	0	5	28.35 ± 1.12	757.09 ± 28.08
A-4	280	0	3	36.38 ± 1.03	795.38 ± 20.98
A-5	250	50	5	30.48 ± 1.87	807.08 ± 25.75
A-6	250	50	3	46.27 ± 2.08	857.67 ± 31.54
A-7	250	0	5	27.24 ± 1.18	702.84 ± 21.53
A-8	250	0	3	38.07 ± 1.25	786.57 ± 24.87
A-O	280	100	2	68.66 ± 2.74	987.46 ± 37.57

Table 2. Results of curve fitting of AC-containing GG-APPM

Model of Kinetics	**R^2 values for various Formulations**								
	A-1	**A-2**	**A-3**	**A-4**	**A-5**	**A-6**	**A-7**	**A-8**	**A-O**
Zero order	0.97751	**0.99004**	0.97999	**0.99334**	**0.99466**	**0.98804**	0.97759	**0.99921**	**0.98867**
First order	**0.98891**	0.95007	**0.98809**	0.95772	0.92777	0.92222	**0.99919**	0.97720	0.97743
Higuchi	0.66883	0.74557	0.72229	0.77644	0.75155	0.71997	0.72227	0.77770	0.78813
Korsmeyer-Peppas	0.93309	0.97795	0.94111	0.81099	**0.99770**	**0.98865**	0.92880	0.97744	0.95990
	Diffusion Exponent (n)								
	0.84111	0.85554	0.79993	0.97782	0.89962	0.92222	0.76558	0.78119	0.74115

R^2 = *Squared correlation coefficients*