



# Profitable exploitation of biodegradable polymer including chitosan blended potato peels' starch waste as an alternative source of petroleum plastics

Tarek H. Taha<sup>1</sup> · M. A. Abu-Saied<sup>2</sup> · Mohamed Elnouby<sup>3</sup> · Mohamed Hashem<sup>4,5</sup> · Saad Alamri<sup>4,6</sup> · E. A. El Desouky<sup>7</sup> · Kareem Morsy<sup>4,8</sup>

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## Abstract

Humans are extensively using synthetic plastic products for multiple life purposes. Non-degradability of these products affected the nature and extremely harms the environment. Herein, biodegradable membranes with different ratios of chitosan/starch waste have been prepared. These membranes have been submitted for in vitro enzymatic biodegradation followed by examination by SEM, FTIR, Raman scattering spectral analyses, surface roughness measurement, degree of swelling, and contact angle. The obtained membranes showed biodegradability significant features which increased by increasing starch ratio and has been confirmed by the formation of holes that were demonstrated by SEM micrographs. Both of surface roughness and water uptake values of the prepared membranes have been increased in all prepared membranes after the enzymatic treatment to the maximum measured value of 0.87  $\mu\text{m}$  and 200%, respectively. However, water contact angle values of the prepared membranes have been decreased after the enzymatic treatment to reach 25  $\theta$  as the lowest measured value. These results suggest the application of chitosan-blended starch membranes as alternatives for petroleum plastics with more environmental safety.

**Keywords** Chitosan · Potato peels' starch waste · Biodegradable plastic · Amylase enzyme · Environmental safety

## 1 Introduction

Plastic products are extensively used in various applications and daily life purposes. Although there are a lot of advantages to plastic applications, their disadvantages are increasing obviously.

Recently, plastic waste is one of the most-faced issues over the world both for environmental problems and human health hazards. Most of the plastics derived from fossil resources are largely non-biodegradable and harmful to the environment and marine life [1]. So, alternative and sustainable solutions are highly

✉ Tarek H. Taha  
t.h.taha@gmail.com

✉ M. A. Abu-Saied  
mouhamedabdelrehem@yahoo.com

<sup>1</sup> Environmental Biotechnology Department, Genetic Engineering and Biotechnology Research Institute (GEBRI), City of Scientific Research and Technological Applications (SRTA-City), New Borg El-Arab City, Alexandria 21934, Egypt

<sup>2</sup> Polymer Materials Research Department, Advanced Technology and New Materials Research Institute, City of Scientific Research and Technological Applications (SRTA-City), New Borg El-Arab City, Alexandria 21934, Egypt

<sup>3</sup> Composite and Nanostructured Materials Research Department, Advanced Technology and New Materials Research Institute, City of Scientific Research

and Technological Applications (SRTA-City), New Borg El-Arab City, Alexandria 21934, Egypt

<sup>4</sup> College of Science, Department of Biology, King Khalid University, Abha 61413, Saudi Arabia

<sup>5</sup> Faculty of Science, Botany and Microbiology Department, Assiut University, Assiut 71516, Egypt

<sup>6</sup> Prince Sultan Ben Abdulaziz Center for Environmental and Tourism Research and Studies, King Khalid University, Abha, Saudi Arabia

<sup>7</sup> Chemistry Department, Faculty of Science, Alexandria University, Alexandria, Egypt

<sup>8</sup> Zoology Department, Faculty of Science, Cairo University, Cairo, Egypt

required to address this issue. Many efforts have been done to reuse and recycle the huge amount of human plastic wastes.

Unlike conventional plastics which are derived from fossil oil; bio-based plastics are derived from renewable resources. Bioplastics are currently more demandable than chemically synthesized plastics and are considered promising alternatives [2]. They could be fabricated from various bioresources and wastes, including food waste, agriculture waste, wastes from animal sources, and alternative sources such as cellulose, protein, and starch [3, 4]. They could be used in numerous application fields, such as food packaging, medical care, horticulture [5], agriculture [6], electronics [7], diapers, and cutlery with great using potentials in the future [8]. They can be defined as naturally produced plastic that originating from renewable sources such as sugar, corn, and potato [9, 10] and can be produced by a wide range of microorganisms [11]. They have the advantages of being bio-based, biodegradable, photodegradable, and compostable. Being biodegradable means that these bioplastics can be naturally disintegrated and broke down into carbon dioxide and water by the action of environmental microorganisms under humidity conditions [12].

Bioplastics from renewable resources are mostly cheaper than those produced by microbial resources [13, 14], which led manufacturers to focus on the production of bioplastics from renewable resources. Starch is considered a promising material produced by renewable resources that can be applied as a bioplastic because of its inexpensiveness and availability [13, 15, 16]. It is already used in different industrial areas such as pharmaceuticals, textiles, paper, and food industry [17, 18], but is expected to be adopted by many companies for the production of bioplastics.

In recent years, increasing interest in membranes has developed mainly due to concern over the disposal of conventional synthetic plastic materials derived from petroleum. Degradation of plastics requires a long time and most of them end up over-burdening on landfills. Conversely, membranes from renewable agriculture products not only are degraded readily after their disposal but also can extend the food shelf-life, thus improving the quality of food. Among various available membrane materials, considerable attention has been given to chitosan because of its unique properties.

Chitosan is derived from chitin, which is the second most abundant polysaccharide on earth next to cellulose and is available from waste products in the shellfish industry [19, 20]. Starch has been used to produce biodegradable films to partially or entirely replace plastic polymers but limited by its water solubility and brittleness [21]. To overcome these shortcomings, Jagannath et al. [22] blended starch with different proteins to decrease the water vapor permeability of the films and to increase their tensile strength (TS).

This work aimed to valorize the exploitation of starch that is extracted from potato peels to be used in the preparation of bioplastic. The extracted starch was mixed in different proportions with chitosan polymer followed by characterization of the prepared membranes using SEM, FTIR, Raman spectroscopy, surface roughness, water uptake, and contact angle. The prepared membranes were submitted to enzymatic treatment to test their biodegradability.

## 2 Materials and methods

### 2.1 Materials

Chitosan (CTS) (M.wt: 100,000–300,000) was obtained from across organics, Belgium. Acetic acid was obtained from Doummar Company, Germany. The  $\alpha$ -amylase enzyme was purchased from Sigma Aldrich.

### 2.2 Starch extraction

Potato peels were collected from households and restaurants wastes at Borg El-arab city, Alexandria. The collected potato peels were cut into small pieces using a kitchen scissor. A volume of 100-ml water was added to each 50 g of the potato peel pieces followed by blending using an electrical blender. The mixture was passed through steel mesh to allow the soften mix to pass and prevent the solid unblended matrix. The collected filtrate was transferred to a glass beaker and was dried in a dry oven at 60 °C for 2 days. After complete dryness, the obtained white powder was scratched, ground into small particles, and kept at room temperature till use (Fig. 1). It would be mentioned that the starch particles were grinded into fine particles to avoid agglomeration during use.

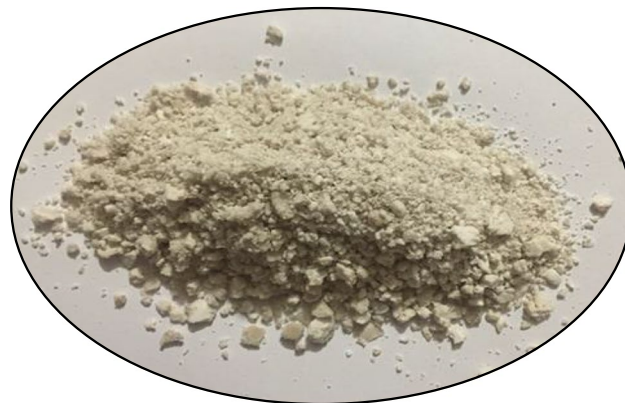


Fig. 1 Potato peels' starch after extraction and dryness

## 2.3 Preparation of chitosan and CTS/starch-based membranes

CTS solution (2%) was prepared by dissolving CTS in an aqueous acetic acid solution (2%). The starch solution was prepared by dispersing varying proportions of starch in distilled water (1–4%). Blended membrane-forming solutions were prepared by mixing their pre-prepared solutions with stirring at room temperature overnight to obtain CTS/starch blends at different ratios of 2:1, 1:1, and 1:2 at weight basis [23]. The resultant blend solutions were cast in plastic Petri dishes and kept at room temperature until full dryness, then the membranes were cross-linked using 5 M NaOH for 24 h followed by washing using distilled water.

## 2.4 Characterization of membranes properties

### 2.4.1 FTIR and Raman scattering spectral analyses

Both of enzymatically treated and untreated CTS and CTS/starch-based membranes were investigated using FTIR (Shimadzu FTIR-8400 S, Japan) and Raman scattering spectrometer (SENTERRA-Bruker, Germany) in order to investigate the chemical structure of the polymers and explore their potential interactions. “All membranes were also scanned using FT-IR spectrophotometer (Model, 8400 s Shimadzu, Kyoto, Japan) over 4000–400  $\text{cm}^{-1}$  with S/N ratio of 30,000:1. Both background scan and sample scan were sequentially measured at a resolution of 4  $\text{cm}^{-1}$  and 2.8 mm/s mirror speed for a total of 10 scans” [24–26].

### 2.4.2 Scanning electron microscopic analysis

Morphological features and microstructure of polymer-based membranes before and after the biodegradability assay were examined using SEM (JEOL JSM-6360LA, Japan). Specimens of the resultant membranes were examined at an acceleration voltage of 10 kV using a magnification power from 500 to 5000 [27–29].

### 2.4.3 Surface roughness measurement

The surface roughness was determined by surface roughness meter (SJ-201P, Japan). Samples of the prepared membranes, before and after enzymatic treatments, were mounted on a glass slide with a double-sided tap. All results were recorded as an average of ten measurements.

### 2.4.4 Degree of swelling and contact angle

Water uptake (WU) or degree of swelling of the membranes was determined. A previously dried membrane sample ( $W_d$ ) was weighed and immersed in distilled water at

room temperature. After 24 h, the sample was taken from the aqueous medium, dried slightly to remove excess water, and reweighed ( $W_w$ ). The degree of swelling was calculated by the following equation [30–32].

$$WU = \frac{W_w - W_d}{W_d} \times 100$$

The contact angle ( $\theta$ ) of a water droplet with the prepared membranes was measured before and after enzymatic treatment using a Drop Shape Analysis System (Ramé-Hart Instrument Goniometer, model 500-F1, France).

### 2.4.5 Tensile measurement

Measurements of specimens with 50 × 10 mm were carried out at a constant speed of cross heads movement 5 mm/min using Universal Testing Machine (Shimadzu UTM, Japan). Samples were measured three times and the mean values were considered [33, 34].

### 2.4.6 In vitro testing of membranes biodegradability

To test the biodegradability of the prepared membranes, a fixed weight (0.03 g) of each membrane was added to an eppendorf tube followed by the addition of 1400  $\mu\text{l}$  acetate buffer (pH 5.5) and 100  $\mu\text{l}$  of amylase enzyme (500 units/g). All the tubes were incubated at 30 °C and 150 rpm for 72 h. After incubation, the concentration of the released glucose units was determined using glucose kit (Bio-System, Spain). Moreover, the instrumental analysis including SEM, Raman spectroscopy, and FTIR analysis were investigated using the solid residuals of each degraded membrane after dryness.

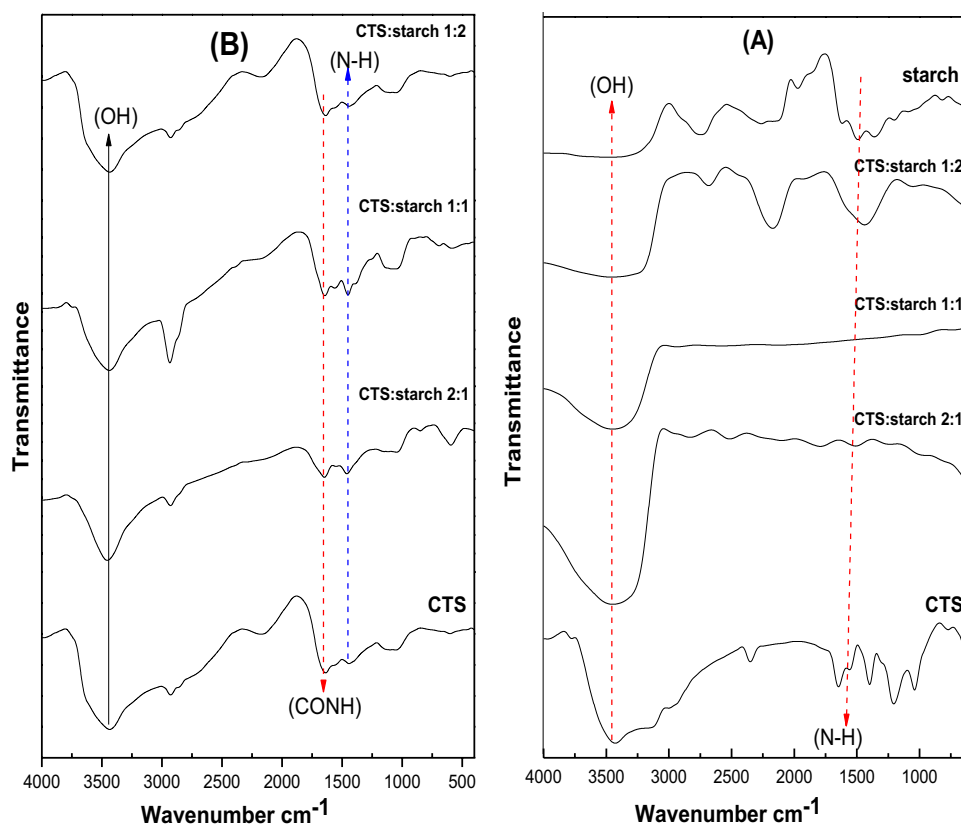
## 3 Results and discussion

### 3.1 Characterization

#### 3.1.1 FTIR spectra

FTIR spectrum of pristine chitosan shown in Fig. 2 revealed the characteristic peak of N–H at 1578  $\text{cm}^{-1}$  in addition to another peak at 1259  $\text{cm}^{-1}$  that is due to N–CH<sub>3</sub> stretching. Additionally, The FTIR spectra of starch typically show an extremely broadband at 3387  $\text{cm}^{-1}$  and the band at 2930  $\text{cm}^{-1}$  was attributed to the O–H stretching and the C–H stretching vibrations, respectively [35]. Meanwhile, in the spectrum of CTS/starch-blended membranes, the amino peak of chitosan shifted from 1578 to 1584  $\text{cm}^{-1}$  with the addition of starch. This result indicates that interactions were present between the hydroxyl groups of starch and the

**Fig. 2** FTIR spectra of CTS, starch and CTS/starch-blended membranes before enzymatic treatment (A) and after enzymatic treatment (B)



amino groups of chitosan [36]. With the incorporation of the enzyme, the spectrum of the membrane was different than in chitosan–starch blank, especially for a shift at specific wavelengths. The shifts were in the wavelengths at approximately  $2924\text{ cm}^{-1}$  (C–H stretching) instead of  $2930\text{ cm}^{-1}$ ,  $1642\text{ cm}^{-1}$  (C=O stretching of secondary amide), and  $1080\text{ cm}^{-1}$  (C–O–C stretching of cyclic ether) which may be explained by the interaction of  $\alpha$ -amylase with chitosan. Additionally, the intensity of the peak at  $1150\text{--}1040\text{ cm}^{-1}$  was decreased, indicating the action of amylase in cleaving the glycosidic linkages of starch. The effect of enzyme incorporation on the CTS/starch could be confirmed by the presence of characteristics peaks at  $1665\text{ cm}^{-1}$ , indicating presence of an amide group (CONH) [37].

### 3.1.2 Raman scattering spectra

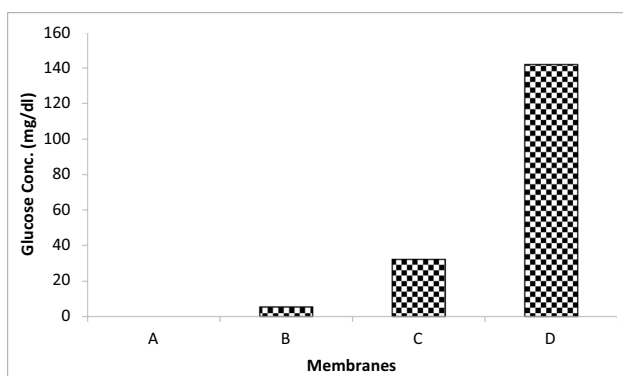
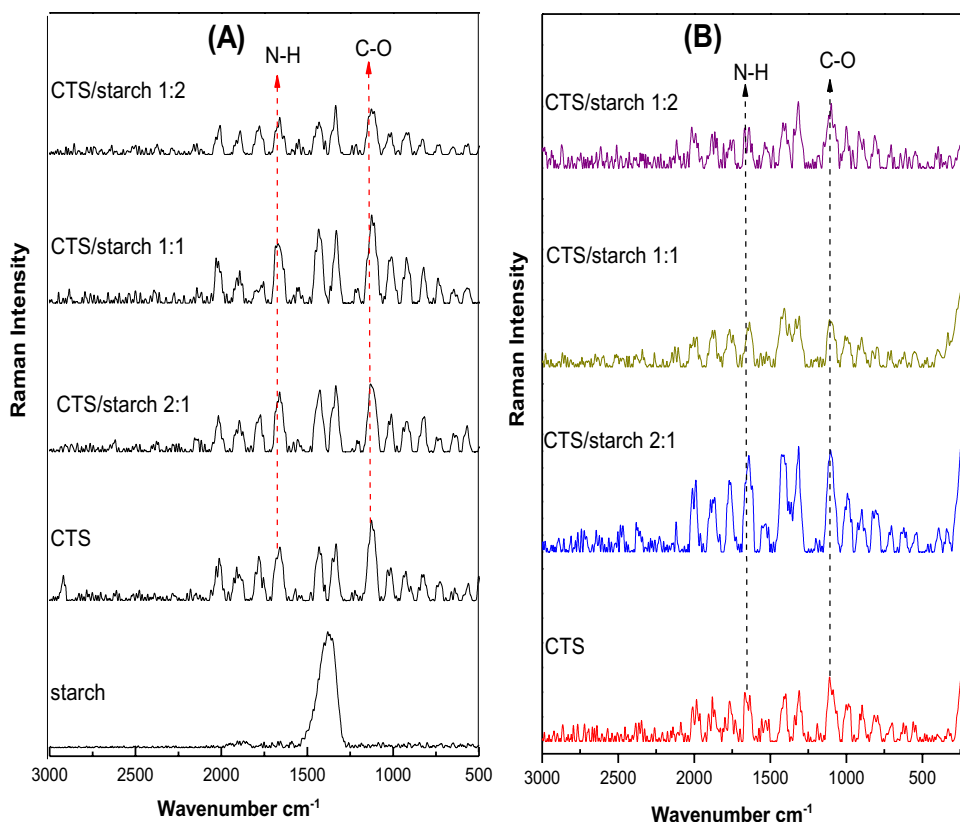
Raman scattering spectrum of pristine chitosan membrane is shown in Fig. 3. This spectrum exhibited a strong valence band at  $1656\text{ cm}^{-1}$  due to C=O bond. Also, CTS spectrum revealed signals at  $1433.19\text{ cm}^{-1}$ , which are attributed to vibrations of N–H, CN band at  $1238\text{ cm}^{-1}$  and C–O band appeared at  $1159\text{ cm}^{-1}$  present into the CTS chains. On the other side, Raman scattering spectrum of starch exhibited three characteristic scattering peaks at  $1263$ ,  $1053$ , and  $941\text{ cm}^{-1}$  which are due to the stretching of C–O, C–C, and

C–OH bonds belonging to starch, respectively. The CTS/starch-based membrane was investigated to find out the molecular interactions between chitosan and starch. Not shifted amide peaks appeared at  $1433\text{ cm}^{-1}$  [35, 38]. The stretching C–N vibration peak appeared at  $1236\text{ cm}^{-1}$  that slightly lowered than the peak of pure chitosan at  $1238\text{ cm}^{-1}$ . While the stretching C–O band came at  $1161\text{ cm}^{-1}$  was slightly higher than pure chitosan ( $1159\text{ cm}^{-1}$ ). Based on the present data, it is noticeably that chitosan was chemically reacted with starch, as expected. Control membranes did not expose any significant differences in Raman bands after enzymatic treatment. However, drastic changes were detected decreases at  $1640$  and  $1564\text{ cm}^{-1}$  from –NHCH–groups (secondary amine) with increasing starch ratio, indicating the action of amylase enzyme in cleaving the glycosidic linkages of starch [39].

### 3.2 In vitro biodegradability test

The biodegradability of CTS/starch membranes with the three prepared ratio (1:1, 2:1, and 1:2) was investigated through the incubation of individual pieces of the membranes with amylase enzyme in acetate buffer solution. The biodegradability was indirectly investigated through the formation of glucose units, which in turn is matched with the biodegradability ratio and reflects the ability of amylase

**Fig. 3** Raman scattering spectra of CTS, starch and CTS/starch blended membranes before (A) and after (B) treatment with amylase enzyme



**Fig. 4** Concentration of glucose units released through the biodegradation of CTS/starch membranes using amylase enzyme. (A): plain chitosan, (B): chitosan: starch (1:1), (C): chitosan: starch (2:1), and (D): chitosan: starch (1:2)

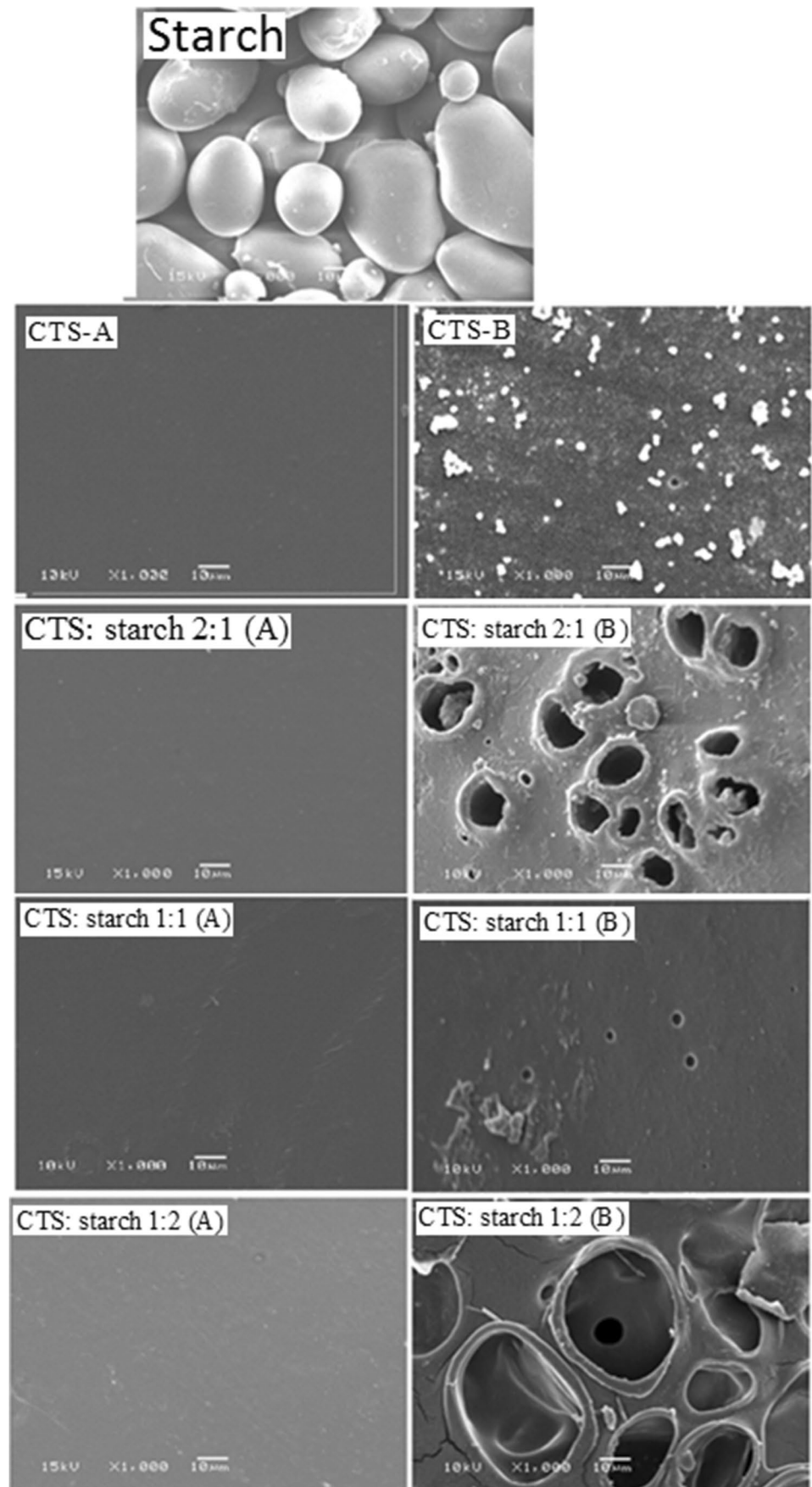
enzyme to degrade the membrane and releasing one of its structural initial units. The three tested membranes showed varying amounts of glucose which are matched with the concentration of the starch molecules (Fig. 4). At ratio 1:1 of chitosan: starch, a lower percentage of glucose units (5.4 mg/dl) was released compared with a moderate and higher percentage of glucose units releasing from the other tested chitosan: starch ratio (2:1 and 1:2). The measured glucose concentrations were recorded as 32.3 and 142 mg/dl, respectively. These results revealed that the film of chitosan:

starch (2:1) showed lower biodegradability than the film of chitosan: starch (1:2) which indicates that the starch percentage is significantly affect the biodegradation process. On the other hand, the film with a (2:1) chitosan: starch showed greater biodegradability during the enzymatic process than 1:1 ratio film. We could attribute this phenomenon to the formation of interactions between the hydroxyl groups of starch and the amino groups of chitosan which might decrease the biodegradability of the membrane especially with increasing concentrations of chitosan (as explained in FTIR results in Fig. 2). This is also matched with the fact that the used amylase enzyme can perfectly and specifically degrade starch molecules and this degradation ability will certainly decrease if starch molecules interacted with other structures such as chitosan. Some researchers depended in testing the biodegradability of starch-based plastic through soil burying, which is almost depending on the activity of the soil microorganisms [2]. However, using of already excreted and purified enzymes is much preferred due to their greater catalytic activity and the absence of seasonal fluctuations [40].

### 3.3 SEM micrograph

SEM microphotographs of starch granules, as well as the surface of the CTS/starch membranes, are shown in Fig. 5 A and B. The starch granules showed large, oval, with compact and smooth surfaces [41]. On the other hand,

**Fig. 5** SEM micrograph of the surface of starch, CTS and CTS/starch blend-based membranes before (A) and after (B) amylase treatment at magnification power of 1000 $\times$



**Table 1** Surface roughness values of CTS/starch membranes before (A) and after (B) enzymatic treatment

Sample	(A) Roughness ( $\mu\text{m}$ )	(B) Roughness ( $\mu\text{m}$ )
CTS	$0.19 \pm 0.02$	$0.40 \pm 0.05$
CTS: starch 2:1	$0.29 \pm 0.05$	$0.47 \pm 0.07$
CTS: starch 1:1	$0.42 \pm 0.04$	$0.56 \pm 0.05$
CTS: starch 1:2	$0.50 \pm 0.03$	$0.87 \pm 0.08$

the surface of chitosan membranes appeared homogenous, smoother, and denser with no gross defects. The observed smooth and homogenous surface of the membranes is an indicator of the structural integrity of the observed membranes [42, 43]. It also indicated enhanced solubilization and homogenization of chitosan in the aqueous medium. CTS/starch membranes showed a homogeneous surface free of porous or cracks and without phase separation between the polysaccharides, which is an indication of good interaction between the components [20, 44]. After enzymatic treatment, it is possible to observe that the prepared membranes exhibit rougher and pored surfaces (Fig. 5B). Also, SEM micrographs show fractures on the surface of CTS/starch membranes due to the removal of starch from the polymeric blend prepared membranes [45]. These observations are matched with the biodegradability data which confirms the ability of the amylase enzyme to degrade the prepared membrane releasing simple glucose units instead of the already existed polymeric matrix. These observations are also in agreement with the data of FTIR and Raman analysis which proved the appearance of new functional groups after the enzymatic treatment indicating the breakdown of polymeric internal bonds [46–49].

### 3.4 Surface roughness

Surface roughness values of CTS, CTS/starch membranes before and after enzymatic treatment are shown in Table 1. Roughness of the surface of plain CTS membrane was the lowest value ( $0.19 \mu\text{m}$ ). Such value was affected by blending CTS with starch, especially CTS/starch (1:2) which showed the highest value ( $0.50 \mu\text{m}$ ). After enzymatic treatment, in CTS/starch membranes, the surface became rougher which indicate that the enzyme was completely incorporated into the polymer network [45], in addition to its ability to degrade the starch material resulting in the formation of internal pores which in turn increasing the surface roughness of the treated membranes. These data are completely matched with SEM micrographs and biodegradability test of the enzymatic treated membranes as shown in Figs. 4 and 5.

**Table 2** Water Uptake values of CTS/starch membranes before (A) and after (B) enzymatic treatment

Sample	WU% (A)	WU% (B)
CTS	$35 \pm 4$	$50 \pm 5$
CTS: starch 2:1	$60 \pm 2$	$80 \pm 3$
CTS: starch 1:1	$70 \pm 4$	$150 \pm 2$
CTS: starch 1:2	$100 \pm 5$	$200 \pm 4$

**Table 3** Water contact angle values of CTS/starch membranes before (A) and after (B) enzymatic treatment

Sample	Mean theta $\theta$ (A)	Mean theta $\theta$ (B)
CTS	$70 \pm 4$	$60 \pm 5$
CTS: starch 2:1	$65 \pm 2$	$40 \pm 3$
CTS: starch 1:1	$60 \pm 4$	$30 \pm 2$
CTS: starch 1:2	$50 \pm 5$	$25 \pm 4$

**Table 4** Mechanical properties of CTS and CTS/starch membranes

Sample	Tensile strength (MPa)	Elongation at break (mm)
CTS	$53.12 \pm 2$	$1.34 \pm 0.2$
CTS: starch (2:1)	$50.14 \pm 1$	$2.11 \pm 0.3$
CTS: starch (1:1)	$45.51 \pm 3$	$4.01 \pm 0.5$
CTS: starch (1:2)	$33.82 \pm 1$	$6.80 \pm 1$

### 3.5 Degree of swelling and contact angle

The swelling ratio of CTS/starch membranes was increased gradually with increasing starch ratio. As shown in Table 2, the pristine chitosan stabilized by NaOH treatment showed the lowest swelling ratio (around 35%), which is much lower than the starch conjugated counterparts that were changed between 60 and 100%. After enzymatic treatment, the swelling degree was increased to 200% for CTS: starch (1:2) membrane. The hydrophilic/hydrophobic nature of a membrane surface is often indicated by the contact angle. Water contact angle values of prepared membranes are listed in Table 3. Generally, the contact angle of CTS membranes was decreased with the addition of starch. This decrease was more pronounced after enzymatic treatment [50].

### 3.6 Tensile strength

The tensile strength and elongation at break for the prepared membranes are given in Table 4. When starch concentration in blend formulation was increased, the tensile strength decreased from 53.12 of plain CTS to 33.82 MPa at CTS/starch (1:2), while the elongation was increased from 1.34 to 6.80 mm,

respectively. These data indicate that the blended CTS/starch (1:2) film has appropriate mechanical properties to be used as alternatives for petroleum plastics with more environmental safety.

## 4 Conclusion

Advanced biodegradable plastic has been synthesized from chitosan and potato peels' starch waste. The obtained membranes showed significant biodegradability features which have been approved by different characterization methods. FTIR and Raman spectral analyses showed the biodegradability of the synthesized plastic before and after enzymatic treatment. According to the obtained results, the chitosan/starch (1:2) blend successfully increases the swelling properties, the elongation, and finally showed a higher biodegradation capacity, which would be recommended as the optimum blending percentage between the two polymers regrading using as alternative source of petroleum plastics, which is highly matched with the current research idea. These films represented higher mechanical properties and more rapid decomposition. Therefore, in our opinion, and according to the obtained results, this research was succeeded to develop bioplastic films that could be considered as a biodegradable alternative source of petroleum plastics using organic material from starch waste combined with chitosan as composites. Additionally, this study concluded that the prepared biodegradable plastic could be used instead of synthetic plastic products in various daily life applications as future eco-friendly materials.

**Author Contribution** Tarek H. Taha: conceptualization, methodology, investigation, formal analysis, data curation, writing—original draft, writing—review and editing. M. A. Abu-Saied: conceptualization, methodology, investigation, formal analysis, data curation, writing—original draft, writing—review and editing. Mohamed Elnouby: methodology, investigation, formal analysis, writing—review and editing. Mohamed Hashem: formal analysis, writing—original draft, writing—review and editing. Saad Alamri: formal analysis, writing—review and editing. E. A. El Desouky: methodology.

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## Declarations

**Conflict of interest** The authors declare no competing interests.

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