



REVOLUTIONIZING WASTE COOKING OIL: ENHANCING QUALITY AND PHYSICOCHEMICAL PROPERTIES THROUGH SILICA EMBEDDED CHITOSAN MEMBRANE

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Abstract: Prior research indicates that chitosan membranes exhibit a highly rigid and non-porous morphology, limiting their utility in filtration applications. To enhance the performance characteristics of chitosan membranes, modification strategies are necessary. Incorporation of amorphous silica into chitosan membranes presents a potential solution to address deficiencies in their physical and mechanical attributes. The objective of this investigation is to evaluate the impact of amorphous silica content on the physicochemical properties of chitosan-silica composite membranes derived from rice husk (RH) by acid hydrolysis. Acid treatment routes produce amorphous silica particles with high purity and more controlled structural properties as compared to other routes like open burning. The membranes were characterized for their morphological, functional group and thermal properties, ensuring their suitability for the purification process. The experimental setup involved the filtration of used cooking oil through the silica-chitosan membrane, targeting the removal of contaminants such as free fatty acid, water, and particulate matter. The results show a substantial reduction in impurity content. This study contributes to the development of sustainable and cost-effective methods for purifying waste cooking oil, making it a viable feedstock for biodiesel production.

Keywords: Biodiesel, Purification, Rice husk, Silica-chitosan membrane, Waste cooking oil.

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INTRODUCTION

Waste cooking oils (WCOs) is the oil that has been used in frying multiple times and categorized as waste. As frying is the most common cooking method used worldwide, WCOs are created in

large quantities and are distributed across the globe. The European Union (EU) contributes around 1 million tonnes annually to the global production of used vegetable oils, which surpasses 190 million metric tonnes annually



(Lin *et al.*, 2013; Mannu *et al.*, 2020; Khanal and Shah, 2021). Dumping of it can cause environmental damage and many harmful diseases. Those who cook and consume fried meals made with used cooking oil have a higher chance of high blood pressure and occurrence of cancer than persons who change their cooking oil regularly (Zhang *et al.*, 2019; Adnyana *et al.*, 2023).

Cooking oil is a mixture of long chain free fatty acids (mainly linolenic acid, linoleic acid, oleic acid) in form of tri, di and mono glycerides, and some percentage of free fatty acids (Avagyan and Singh, 2019; Liu *et al.*, 2020). During frying process, some portion of triglycerides of ester moiety breaks down and generates many volatile compounds as a consequence of combination between increasing temperature and oxygen addition, which promotes oxidation and other chemical transformation (Ziaififar *et al.*, 2008; Singhabandhu and Tezuka, 2010). Some physical observations in oil quality can be observed with its unpleasant odor, darker or less clear color and high density. There are many parameters to indicate the oil quality like free fatty acid value, peroxide number and acid value (Rusdi *et al.*, 2021). The goal of purifying WCOs is to break down any unwanted ingredients while preserving the oil's unique qualities. There are two methods for physically purifying WCOs. First treatment method includes filtering, distillation, and extraction. The second treatment method involves chemical transformation to exploit the chemical functional groups present in the waste for the synthesis of added value products (Mannu *et al.*, 2020).

A most promising method to purify WCOs is membrane separation technology. Only fewer amounts of chemicals are used in membrane technology as compared to other process. Along with this, membrane technology allows selective separation of different components in WCOs with specific pore size designing. Membrane process often requires less energy as compared to other methods like distillation and chemical method (Yadav *et al.*, 2022). Some researchers have modified membrane by adding material and polymer for better character of the membrane like increase in stability and reduced pores size to

make the separation more efficient (Liu and Bai, 2005; Soares *et al.*, 2017; Dakroury *et al.*, 2020; Ibrahim *et al.*, 2022; Pandis *et al.*, 2022).

A membrane that can be modified with a mixture of natural sources like silica extracted from waste biomass i.e. rice husk (RH) may be an eco-friendly approach and more efficient method to purify WCOs. RH, which is considered as a waste in rice factories can be a very good source of silica extraction as it contains almost 18-20% silica. The fragile and low biocompatibility of silica can be modified by combining it with chitosan. Silica is also able to increase the stability and pores of chitosan membrane (Repo *et al.*, 2011; Riana *et al.*, 2022).

This study contributes to the development of cost-effective root for purifying waste cooking oil, which underscores its potential as a green technology for addressing environmental concerns associated with improper disposal of WCOs. The findings offer valuable insights for researchers, engineers, and industries seeking eco-friendly solutions for biodiesel feedstock production and waste management (Khanal and Shah, 2021).

MATERIALS AND METHODS

Chitosan was purchased from Sigma-Aldrich (chemical sciences company) with average degree of acetylation of up to 94 mol%. Glacial acetic acid (1%) was used as a solvent and dimethyl formamide was used as an additive. H_2SO_4 was used as an extraction solution for silica from RH. KOH, and ethanol were used for acid number analysis. Chloroform, periodic acid, sodium thiosulphate, potassium iodide and distilled water were used in free glycerol analysis. All chemicals of analytical grade were purchased from Sigma-Aldrich. RH was purchased from Amazon online platform.

Silica extraction from RH

RH was washed with distilled water to remove all the dust particles and water-soluble impurities and then dried overnight in a hot air oven. After complete drying overnight in a hot air oven. After complete drying, the washed RH was acid treated with H_2SO_4 . After acid treatment, RH was washed multiple times with distilled water to remove the

sulphate group and then dried to remove all the moisture. The dried pretreated RH was then weighed and poured in an alumina crucible and calcined at 600° in a muffle furnace for 2 hours with increasing temperature 4°/min to obtain highly pure white amorphous silica powder (Fig. 1).

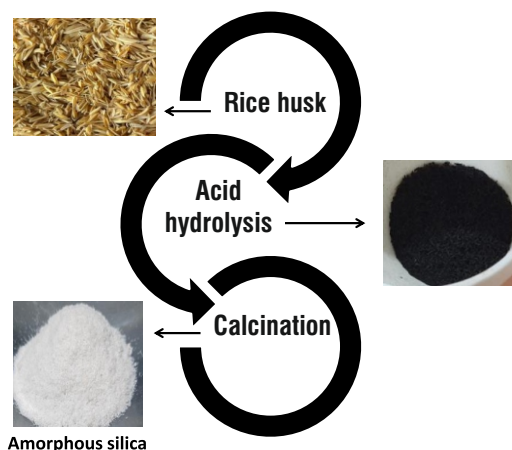


Fig.1: The schematic presentation of the process followed for amorphous silica extraction from rice husk.

Preparation and optimization of membrane

3% (w/v) concentration of chitosan was added into 10% (v/v) dimethylformamide and 1% (w/v) glacial acetic acid. The mixture was continuously stirred at 300 rpm until chitosan was completely mixed in the solution. This same concentration of solution was prepared in triplet. Sample one contains only chitosan and named as CM1. The second sample was added with 3% (w/v) of silica powder obtained from RH and mixed until a homogeneous solution was obtained. (w/v) of silica powder obtained from RH and mixed until a homogeneous solution was obtained. After complete dissolution of silica particles, the solution was printed on a petri plate and cooled down to evaporate the solvent at room temperature to obtain chitosan membrane embedded with silica particles. This sample was named as SCM1. The membranes obtained were washed with NaOH and rinsed with distilled water and then dried at room temperature (Fig. 2).

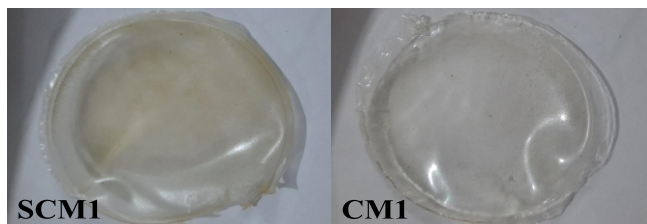


Fig. 2: Images of prepared membranes SCM1 and CM1.

General characterizations of the prepared membranes

Water stability

The prepared membranes were cut into 2.0 m² size and soaked into distilled water at room temperature for 24 hours. The initial dry weight and wet weight of the membrane was measured to check whether the membrane was water soluble or not.

Thickness

The thickness for all the membrane was determined with screw gauge.

Porosity and swelling degree

The porosity and swelling degree of the prepared membrane was determined based on water absorption capacity of membrane. The Equations (1) and (2) used to determine porosity and swelling degree, respectively (Riana *et al.*, 2022).

$$\text{Porosity (\%)} = \frac{V_{\text{wet}} - V_{\text{dry}}}{V_{\text{wet}}} \times 100 \quad (1)$$

$$\text{Swelling degree (\%)} = \frac{V_{\text{wet}} - V_{\text{dry}}}{V_{\text{dry}}} \times 100 \quad (2)$$

Where V_{wet} is volume of membrane after soaking in water and V_{dry} is the initial dry volume of the membrane.

Effects on purification of oil sample with prepared membrane

Batch adsorptive purification

The performance of the prepared membranes was evaluated towards WCOs purification. Three sheets of each membrane were cut into 2.5 m² and weighed. Membranes were soaked into 20mL of waste cooking oil taken into Erlenmeyer flask and put on a magnetic stirrer with continuous stirring for different time periods at 300 rpm. The contact durations of 10, 20, 30 and 40 minutes were used to find the optimum contact time carried out using CM1, SCM1. After each filtration, the membranes were assumed to contain oil impurities. So, it was first washed with distilled water and after that immersed into some organic solvent for further use.

Determination of density, acid value and peroxide value for oil sample

Density

The density of the oil sample was measured before and after filtration with CM1 and SCM1 membrane using hydrometer. The Equation (3) used to find oil sample density is as:

$$\text{Density} = \frac{\text{Mass}}{\text{Volume}} \quad (3)$$

Acid value

The AV was calculated following the method of the American Oil Chemists' Society (AOCS) (Crowe and White, 2001).

Five mL of the oil sample and 10 mL of ethanol were combined, and then three drops of phenolphthalein were added. 0.1 M KOH was used to titrate the mixture until a persistent pink color developed. The following Equation (4) was used to calculate acid numbers (Riana *et al.*, 2022).

$$\text{Acid value} = \frac{56.1 \times N_{\text{KOH}} \times V_{\text{KOH}}}{m} \quad (4)$$

Where N_{KOH} is normality of KOH solution used, V_{KOH} is volume of KOH solution used in titration and m is mass of oil sample used.

Peroxide value

Based on the method of Nuru and Getachew, the PV was measured (Nuru and Getachew, 2021). In a mixture containing 100 mL of chloroform and 50 mL of distilled water, 10 mL of oil sample was added. The mixture was vigorously shaken for 3 to 4 minutes and left until the organic layer and the aqueous layer were separated. The addition of 3 mL of distilled water and 2 mL of periodic acid followed the procedure and the mixture was left for 30 minutes. Then, 0.5 mL of KI was poured into it and titrated with 0.01 N sodium thiosulfate solutions until the brown color faded. After that, the mixture was added with starch until it turned blue. The value of total peroxides level was calculated using Equation (5) (Riana *et al.*, 2022).

$$\text{Peroxide value} = \frac{M(B-C) \times 1000}{m} \quad (5)$$

Where B is amount of sodium thiosulfate used for sample and C is amount of sodium sulfate used for blank, M is molarity of sodium thiosulfate solution and m is the mass of oil sample used.

RESULTS AND DISCUSSION

Structural analysis of RH extracted Silica

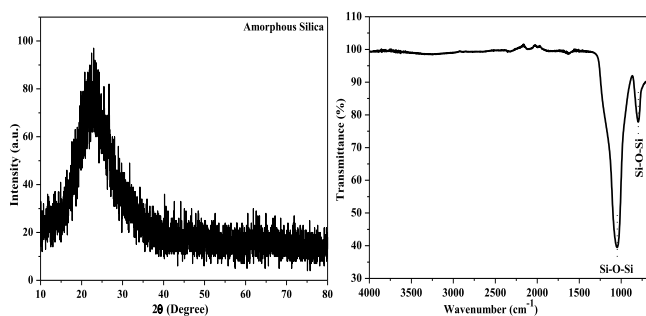


Fig. 3: XRD and FTIR data of RH extracted amorphous silica.

Figure 3 shows the XRD and FTIR spectra of the RH extracted silica. In XRD data there is a presence of hump having width 16 to 30 and center around $2\theta = 22^\circ$ shows amorphous nature of the particles. It is assumed that silica has amorphous like nature (due to large existence of porosity in silica), which confirmed with presence of hump. The absence of any other peak in XRD data indicates the purity of extracted amorphous silica particles. The high intensity of silica peaks and absence of any other peaks in FTIR data also confirms successful extraction of the silica particle with high purity (>94%), as suggested by other studies (Sarangi *et al.*, 2011).

The particle size distribution technique used to find the average particle size of the synthesized silica particles which shows the average particles size was 1406 nm with 0.98 polydispersity index (Figures 4A, 4B, 4C, 4D).

The FE-SEM images shown in Figure 4A indicate the silica particles having a rough surface covered with a spherical structure. It has been observed that its shape is rather irregular, suggesting the possibility of forming a rough surface on the chitosan matrix during preparation for silica embedded chitosan membrane (Figure 4B). The EDS data shows the dominant percentage of Si and O content in the sample (Figures 4C, 4D).

Characterization of Prepared Membranes

Thickness, porosity and swelling degree determination of prepared membranes

The membranes prepared here by adding

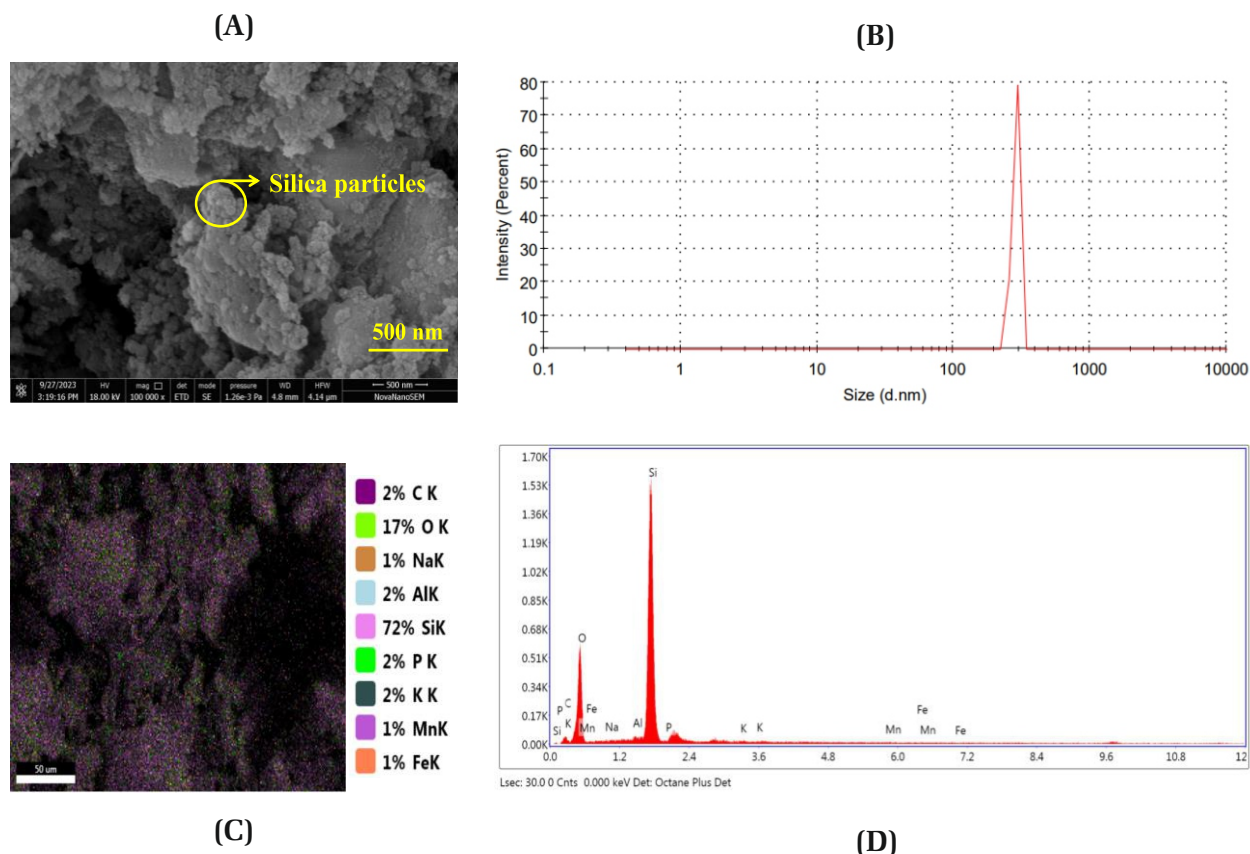


Fig. 4 (A) shows the FE-SEM images, (B) shows the particles size analysis, (C) shows the EDS data mapping and (D) shows the EDS data of silica particles.

amorphous silica and nanocellulose were characterized previously. The prepared pure chitosan and silica-chitosan membrane's thickness, porosity and degree of swelling are presented in Table 1. Compared to silica-chitosan membrane, the pure chitosan membrane has lower thickness, which may be due to the lower

density of silica particles as compared to chitosan. The incorporation of silica nanoparticles also leads to increased cross-linking within the membrane structure, which results in a denser and thicker membrane as compared to the pure chitosan.

Table 1: General characterization of prepared membranes.

Properties	Pure chitosan (CM1)	Silica-chitosan (SCM1)
Thickness (mm)	0.21	0.22
Porosity (%)	35.8	22.8
Swelling degree (%)	41.9	29.7

If the porosity of CM1 is compared with SCM1, it can be observed that SCM1 is less porous than CM1, which may be due to the presence of silica nanoparticles embedded in the chitosan membrane. Silica particles are of nano size, so they can be densely packed. The small size and close packing cause overall reduction in porosity

of SCM1. The porosity and swelling degree of pure chitosan membrane are high due to its hydrophilic nature, which allow more water to absorb. In terms of filler embedment, a lower swelling degree is expected to prevent fillers from leaching out from the membrane.

FTIR data study for prepared membranes

The mixing of two polymers is of great interest of study. To determine the presence of specific functional group and formation of new chemical bonds, FTIR is a very useful tool. If any kind of bonding takes place, it will be shown by presence of peaks at a specific wave number or would be shown as shifting of existing peaks. Figure 5 shows the FTIR spectra for SCM1 and CM1. The FTIR characterization of the prepared membranes showed a broad-spectrum around 3387 cm^{-1} indicating the presence of NH group that overlapped with OH group (Nur *et al.*, 2017). This peak shifted towards lower wavelength from 3307 cm^{-1} to 3284 cm^{-1} in SCM1 spectra, which may be due to the interaction between NH groups of chitosan with OH group of silica (Nur *et al.*, 2017).

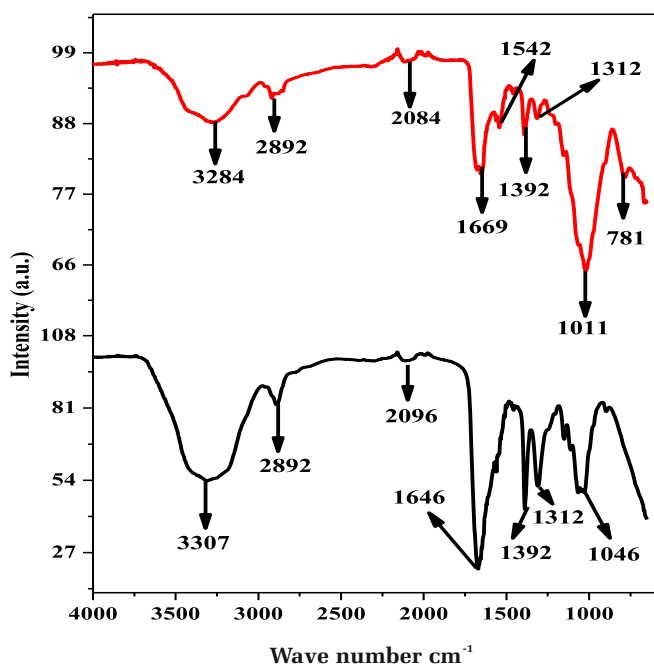


Fig. 5: A comparative FTIR study for SCM1 and CM1 membranes.

Absorption peaks around 2915 cm^{-1} and 2892 cm^{-1} is for CH stretching and a sharp peak at 1646 cm^{-1} present in both spectra are for chitosan moieties, which can be assigned to primary and tertiary amines peaks (Yasmeen *et al.*, 2016). The absorption band present at 1023 cm^{-1} in SCM1 is assigned for Si-O-Si asymmetric stretching vibrations, while new peak at 781 cm^{-1} is for Si-O-C bonding, which is not present in CM1; it

confirms that there is definite interaction between the phases. The shifting of peak from 1046 cm^{-1} to lower wave number 1013 cm^{-1} shows the bonding between silica particles and chitosan molecules. This peak shows the Si-O-Si stretching caused due to bonding of SiO_2 molecule with carbonyl bond of chitosan (Figure 5).

Effect of contact time on physical appearance, density, acid value and peroxide value of oil sample

Physical

The changes in the sample colour after filtration and before filtration are shown in Fig. 6.

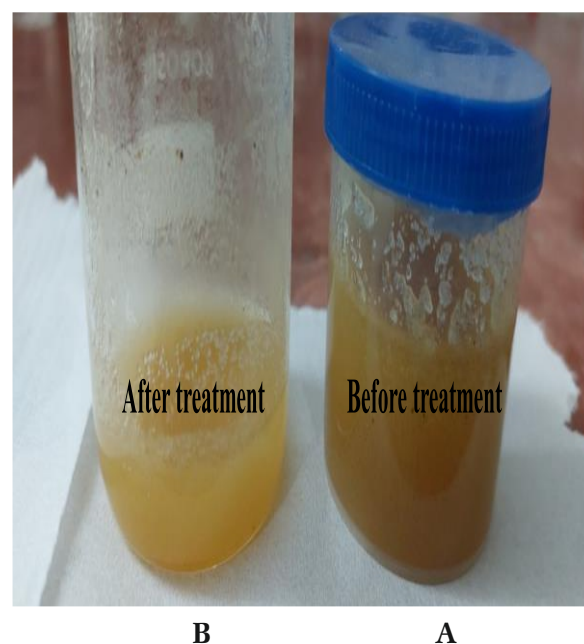


Fig. 6: Images of WCOs before purification (A) and after purification (B).

Density

When silica embedded chitosan membrane came in contact with waste cooking oil the silanol (-Si-OH) groups present on the surface adsorb impurities such as free fatty acids, heavy metals and polar compounds. The combination of silica and chitosan creates a synergistic effect where the silica particles contribute to physical adsorption, while chitosan provides sites for ionic and polar interactions enhancing the overall efficiency of the membrane. Table 2 shows the effect on density of WCOs sample after

filtration with CM1 and SCM1 for different contact time. From the Table 2, it can be confirmed that density for oil sample continuously decreases with increasing contact time but after 20 minutes no such changes were observed. This may be due to over the time; the membrane could become fouled with impurities, reducing its effectiveness. Fouling can clog the pores and reduce the flow rate, preventing efficient purification, and thus leading to minimal change in density. Figure 7 shows the change in oil sample density treated with CM1 and SCM1 membranes for different period of contact time.

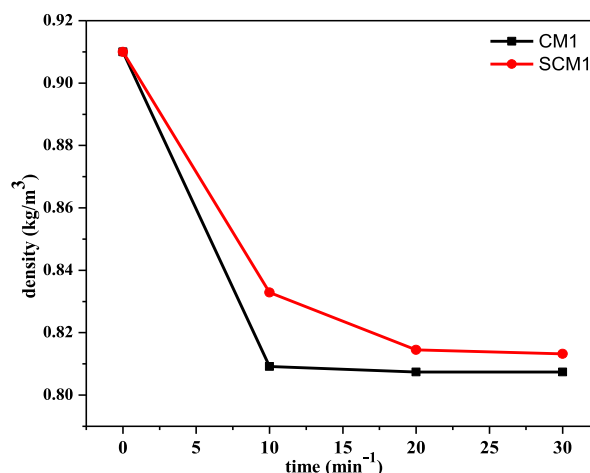


Fig. 7: Effect of contact time on the oil density.

Table 2: Change in oil density after filtration for a particular contact time.

Oil sample (ml)	Contact time (min)	Density (g/ml)	
		CM1	CM1
20	0	0.910	0.910
20	10	0.8092	0.8329
20	20	0.8074	0.8145
20	30	0.8074	0.8132

Acid value

The acid value and peroxide levels in WCOs significantly decreased during the first 20 min (Fig. 8A, 8B), associated with the high diffusion force due to the unoccupied binding site. At this stage, the adsorption was dominated by the physical forces rather than chemical interaction

(Nur and Iqhrammullah, 2020). The decrease in acid number and peroxide content was much less significant and tended to become saturated after reaching 40 minutes. This indicates that at the 40th minute, the binding site of the adsorbent had been fully occupied, and equilibrium of adsorption-desorption occurred.

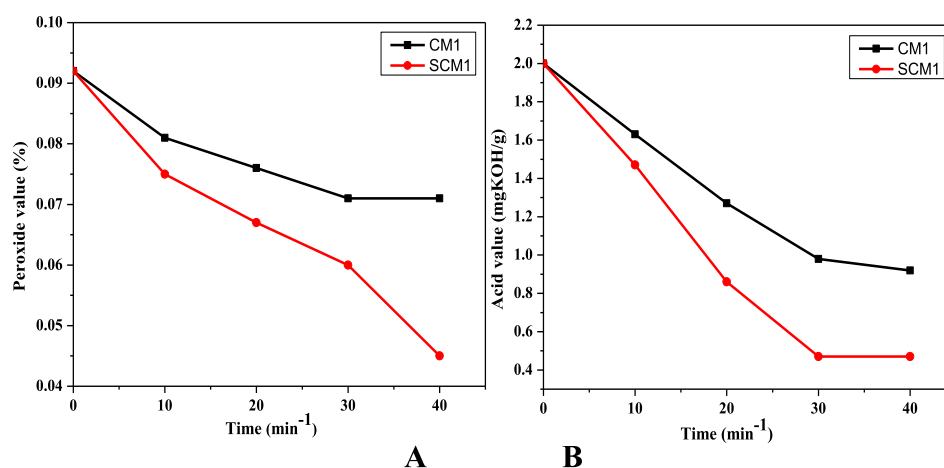


Fig. 8: Effect of contact time on (A) acid value, (B) peroxide value of cooking oil after purification with SCM1 and CM1.

Generally, the acid value for cooking oil should be less than 0.1 mg KOH/g but the waste cooking oil sample taken has acid value 2 mg KOH/g, which reached to 0.47 and 0.92 mg KOH/g after purification with SCM1 and CM1 (Iqhrammullah *et al.*, 2020). The acid value of oil increased with each frying due to hydrolysis reaction. The reason behind increasing peroxide value may be due to reaction of triglycerides present in oil with water, which may produce di-mono glycerides and free fatty acids. The purification with SCM1

shows better results as compared to CM1, which may be due to the presence of silica nanoparticles embedded within chitosan membrane (Table 3). The silica nanoparticles are porous in nature, which is confirmed with the FE-SEM data of silica particles extracted from RH. The porous nature and larger surface area of silica nanoparticles provide higher binding possibility with free fatty acids. This feature improves the adsorption capacity of SCM1 membrane.

Table 3: Acid value and peroxide value of used cooking oil sample before and after purification with CM1 and SCMI membrane.

Oil sample (ml)	Acid value (mgKOH/g)		Peroxide value (%)	
	CM1	SCM1	CM1	SCM1
Without treatment	2.0	2.0	0.092	0.092
10 minutes	1.63	1.47	0.081	0.075
20 minutes	1.27	0.86	0.076	0.067
30 minutes	0.98	0.47	0.071	0.060
40 minutes	0.92	0.47	0.071	0.045

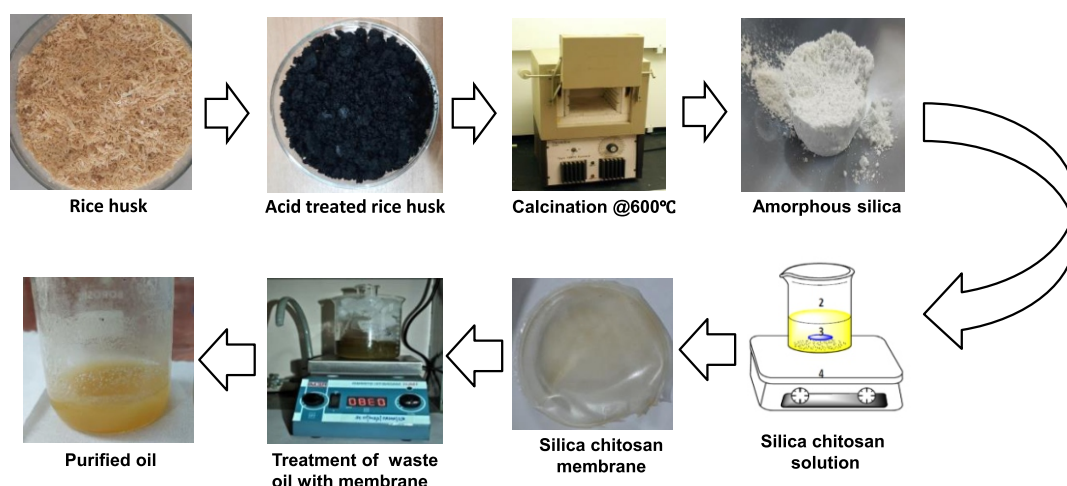


Fig. 9: Graphical abstract of the present work.

The present study revealed that WCOs' adsorptive removal is affected with silica particles loading in chitosan membrane. The change in impurities adsorption is associated with several interactions such as hydrophobic interaction, Vander Waals interaction, chemical bonding or hydrogen bonding. In this case, the SiO_2 molecule may attract the impurity molecules with O-containing moieties through hydrogen bond (Saiful *et al.*,

2018). Due to this interaction, the SiO_2 molecule may also undergo reconstruction and react with water molecule to form Si-OH (silanol group) bonding. This silanol group can also form a complex with water molecules ($\text{SiOH-OH}_2\text{-OH}_2$) (Da Silva *et al.*, 2017). The chitosan matrix may form interactions with the free fatty acid, glycerol, and water molecules that, along with the silica particles, synergistically purify the used

cooking oil sample (Liu *et al.*, 2005; Ebisike *et al.*, 2020). The schematic representation of the present work has been demonstrated in the form of a graphical abstract (Figure 9).

CONCLUSION

RH extracted silica can be considered as a natural adsorbent to purify WCOs. From the data analysis of synthesized silica nanoparticles suggested that it contains almost negligible impurities. Hence, it is safe to use amorphous silica with chitosan for oil purification. Amorphous silica increases adsorption capacity of membrane due to its porous nature, which directly affects oil purification. Amorphous silica particles embedded with chitosan matrix showed supporting properties towards impurities removal from WCOs. From the present study, it can be concluded that performance of SCM1 on impurity adsorption has a significant role with contact time. At optimum contact time the impurities removal successfully reduced the acid number and peroxide value from 2.0 to 1.47 mg KOH/g and 0.092 to 0.075 %, respectively. But after 40 minutes no significant changes were observed in peroxide value and acid value, which may be due to occupancy of binding sites with impurities. Moreover, the color of the sample also improved after purification showing reduction in impurities. This study reveals that RH extracted silica with chitosan can be considered as an effective adsorbent to improve oil quality.

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CONFLICT OF INTEREST

Authors declare that there is no conflict of interest.

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