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Microwave-assisted acid extraction of high-methoxyl kinnow (*Citrus reticulata*) peels pectin: Process, techno-functionality, characterization and life cycle assessment

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ABSTRACT

The present study assessed the efficacy of kinnow peel pectin-acetic acid extraction using microwave heating at 110 °C, pH 2.2 for 10 min with a 1:2 ratio supernatant to ethanol for higher yield. The kinnow peel was freeze dried and grinded to fine powder for pectin extraction. The microwave extracted (ME) kinnow pectin showed 833 mg equivalent weight, 7.44 % methoxyl content, 66.67 % degree of esterification, 63.15 % galacturonic acid content and evinced higher purity than commercial citrus pectin. ME kinnow pectin exhibited shear thinning behaviour while higher apparent viscosity (Pa. s) at 20 % concentration. The ME kinnow pectin showed characteristic functional groups and a less crystalline structure as deduced from FT-IR, SEM and XRD respectively, and a higher thermal decomposition analysed from TGA. Further, life cycle assessment (LCA) predicted that the ethanol and acetic acid were major contributors toward climate change in this study. ME kinnow pectin has the potential to be used as a commercial pectin in various food applications.

1. Introduction

Kinnow is a citrus fruit hybrid developed from two citrus species i.e., *Citrus nobilis* (King) and *Citrus deliciosa* (Willow Leaf). After extracting the juice from kinnow fruit, the residual pomace and peel are discarded as waste, causing environmental pollution due to improper disposal (Purewal & Sandhu, 2020). The kinnow peel contains polysaccharides majorly, pectin and cellulose, along with bioactive such as polyphenols, flavonoids (naringin and limonene), carotenoids, and essential oils which makes it valuable in the pharmaceutical, food and biofuel sectors (Godara et al., 2020). Pectin is an essential ingredient used in confectionary as a thickener and gelling agent and also useful in therapeutic foods or drugs targeted to reduce digestive problems and blood cholesterol levels. In 2019, the worldwide pectin market was estimated to be worth $1 billion while by 2026, the pectin market is expected to develop at a compound annual growth rate (CAGR) of 6.5 % and reach $1.8 billion (Nadar et al., 2022). Commercial pectin is generally produced from citrus fruits; however, it is also present in significant amounts in carrots, sunflower-heads, citrus fruit peel, apple pomace, guava, mango, chickpea husk and papaya as reported by Srivastava and Malviya (2011). The functionality of pectin is dependent on degree of esterification and purity is indicated by the content of anhydrouronic acid (AUA) with a suggested value of at least 65 % for usage as food additives or pharmaceuticals (Khamsucharit et al., 2018; Khubber et al., 2023). In recent years, significant progress has been made in developing innovative techniques for extracting pectin while prioritizing environmental sustainability. The most common green methods explored for pectin extraction include hot water extraction (Ghoshal & Negi, 2020), microwave-assisted extraction (Manzoor & Ahmad, 2021) and Rapid Solid Liquid Dynamic (RSLD) (Benassi et al., 2021; Mahmud et al., 2021). Microwave-assisted extraction (ME), a non-contact heating extraction method has been reported to result higher yield in less processing times with lower production cost for citrus pectin and black carrot (Kumari et al., 2023; Sucheta et al., 2020). Previously, for kinnow peel pectin, the pectin yield for aqueous extraction (18.57 %), acid assisted extraction (20.46 %), microwave assisted extraction (26.87 %) and ultrasound assisted extraction (30.59 %) were compared and concluded that the novel green methods provided better quality and

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yield as compared to conventional methods (Kumari et al., 2023).

Microwave assisted extraction (ME) of pectin depends on critical processing parameters such as time of heating, microwave power, pH, and solid–liquid ratio etc. Prakash Maran et al. (2013) observed that higher microwave power increased pectin yield, while increased time, pH, and solid–liquid ratio reduced it. In another study, microwave-assisted extraction at 15-minute extraction time and a 1:15 solid to acid solution ratio resulted in 51.3 % yield, 10.8 % water content, 5 % ash content, 775.41 equivalent weight and 6.49 methoxyl content of *Citrus sinensis* peel pectin (Wijana et al., 2023).

Previously, Kumari et al. (2023) studied pectin extraction using microwave-assisted extraction from Kinnow peel powder with intermittent heating (900 W, 180 s, 60 s intervals) in 0.05 M HCl with solid-solvent ratio of 1:20, resulting in a pectin yield of 26.87 %. Another study isolated pectin from citrus maxima white pith using acidified conditions using L-(+)-tartaric acid and obtained highest yield (70.2 %) at pH 1.0, 60 °C, 120 min (Daud et al., 2019). However, till date, the study of kinnow peel pectin extraction using organic acids, its characterization, rheology and functionality studies in effect of microwave assisted extraction have not been explored. Further, the lifecycle assessment of the pectin extraction process has not been previously reported in the literature till date.

In view of the above findings, the present study focused on effect of pectin extraction parameters including temperature, pH, time and supernatant-to-solvent ratio using microwave heating extraction (ME). It was done to enhance the yield of pectin from kinnow peels, which was subsequently analysed and compared with commercial citrus pectin for functional properties and characterization studies. The physio-chemical properties of ME kinnow pectin including methoxyl content, degree of esterification and galacturonic acid was observed. The rheological properties of pectin (shear rate vs viscosity) on the basis of power law model, and color values were also studied. The ME kinnow pectin was further characterized for morphology, thermal stability and crystallinity through SEM, FT-IR, TGA and XRD analysis. Further, LCA was carried out for microwave assisted acid-pectin extraction from kinnow peel, to relate the effect of process on the environment. The microwave assisted acid extracted ME kinnow pectin with higher yields and retained functionality, could be a sustainable alternative to citrus pectin for commercialization.

2. Materials and methodology

2.1. Chemicals and materials

Commercially citrus pectin was purchased from Sigma-Aldrich, Germany. All the analytical grade chemicals like ethanol, glacial acetic acid and 0.1 M HCl were purchased from CDH Chemical, New Delhi, India and standards reagents were purchased from Sigma-Aldrich, Germany.

2.2. Plant sample preparation

Kinnow peels were collected from Punjab Agro Juice Industry, Hoshiarpur, Punjab, India and preserved at −20 °C. The peels were then cut into small pieces, kept at −80 °C (Eppendorf, Cryocube 570, Germany) for 24 h, freeze dried at −107 °C in lyophilizer (Coolsafe, Labogene, Denmark), ground to fine powder and packed in air tight container till further use.

2.3. Extraction of pectin from kinnow peel

The pectin was extracted from kinnow peel powder using a microwave heating method and analysed for effect of temperature, time, pH, and supernatant-to-ethanol ratio on pectin yield. To optimize pectin extraction yield, single-factor experiment method was followed. The experiments were designed varying the four factors affecting the pectin yield, including pH (2.0, 2.2, 2.4, 2.6, 2.8 and 3.0), temperature (110, 140, 170 and 200 °C), time (5, 10, 15 and 20 min) and extraction ratio as supernatant: ethanol (1:0.5, 1:1, 1:2, 1:3, 1:4 and 1:5) as shown in Fig. 1. 1 g of Kinnow peel powder was mixed with 100 mL of 1 % acetic acid solution (Sucheta et al., 2020). The extraction solution was maintained to pH 2.2, and then subjected to microwave convective heating (IFB 30SC4, India) at 110 °C for 10 min. The sample was given pulse ratio of t_{on}/t_{off} = 10/10 sec = 1, to minimize the spillage from boiling of solution. The extraction solution was then filtered out using Nexflo filter paper QL grade 1 diameter size 125 mm (Moxcare Labware, India). The supernatant was then dissolved with ethanol in ratio of 1:2 (v/v) (supernatant: ethanol) as shown in Fig. 1 and kept at 25 ± 5 °C for 12 h for precipitation of pectin. Following this, the solution was centrifuged at 6000 rpm at 15 °C for 15 min (Eppendorf, 5910R, Germany) and the pectin residues are washed with ethanol using cell strainer of 100 μm pore size nylon filter and kept for cryopreservation at −80 °C till frozen and lyophilized (Labogene, Coolsafe, Denmark) overnight at-108 °C. The extraction process was carried out in triplicates. The extracted pectin yield was computed using the formula (1) from Muñoz-Almagro et al. (2019).

$$Pectin\ yield\% = \frac{weight\ of\ dried\ pectin\ (g)}{initial\ weight\ of\ sample\ (g)} \times 100 \quad (1)$$

2.4. Physicochemical characterization

2.4.1. Moisture content

An infrared moisture analyser was used to determine the moisture content of kinnow peel powder. (Sartorius MA-35, Germany) at 105 °C for 9.6 min till the constant weight was attained (Mahawar et al., 2017).

2.4.2. Equivalent weight

The equivalent weight was estimated using the modified method of Ranganna (1986) wherein, 0.2 g of powdered pectin sample was mixed in 2 mL ethanol and 20 mL ultrapure water (Integral M3, Merck, USA) and stirred at 50 °C for 30 min at 300 rpm using heating magnetic stirrer (Velp Scientifica, Apex-6 Digital Pro, Italy). The solution was further titrated against 0.1 M NaOH after addition of few drops of phenolphthalein. The equivalent weight of extracted pectin was calculated by formula (2):

$$Equivalent\ weight = \frac{weight\ of\ sample \times 1000}{ml\ of\ alkali \times concentration\ of\ alkali} \quad (2)$$

2.4.3. Methoxyl content (MeO)

The quantity of MeO was determined by adding 25 mL of 0.25 M NaOH to the titration solvent, which was then stirred and kept at room temperature for 30 min (Ranganna, 1986). Then 25 mL of 0.25 M HCl was added and the endpoint was adjusted till appearance of pink colour. The methoxyl concentration was then calculated using the formula (3) below:

$$MeO\% = \frac{ml\ of\ NaOH \times concentration\ of\ alkali \times 31}{weight\ of\ sample} \times 100 \quad (3)$$

wherein, 31 represents methoxyl molecular weight.

2.4.4. Degree of esterification (DE)

A 20 mL pectin solution (0.5 g/100 mL) was prepared, mixed with phenolphthalein reagent and titrated against 0.1 M NaOH solution, until a pink color (V_1) appeared according to the method of Kazemi et al. (2019). Subsequently, 10 mL of 0.1 M NaOH solution was added and stirred for 20 min to intensify the pink color. Afterwards, 10 mL of 0.1 M HCl solution was introduced till the pink color vanishes and then solution was titrated again with 0.1 M NaOH solution to obtain V_2 and DE was estimated using the formula below:

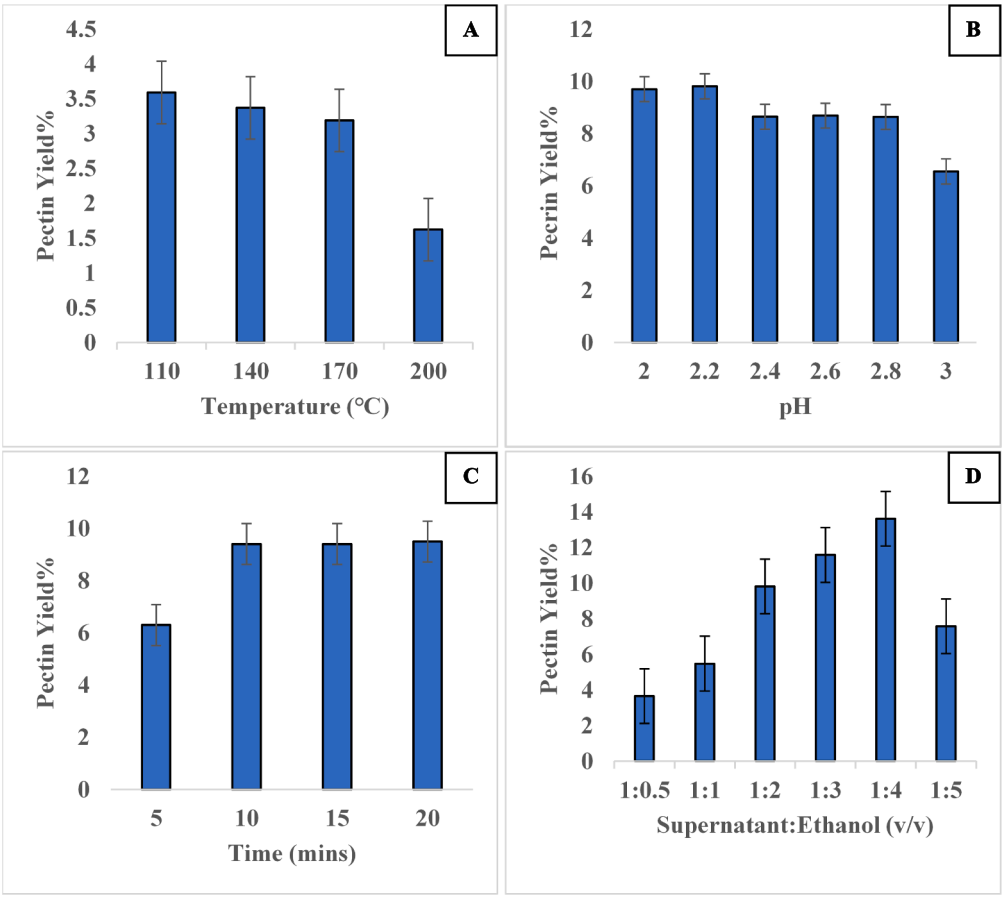

Fig. 1. Effect of different factors on yield of pectin from kinnow peel a) Temperature, b) pH, c) Extraction Time, d) Supernatant to ethanol ratio.

$$DE\% = \frac{V_2}{V_1 + V_2} \times 100 \quad (4)$$

2.4.5. Galacturonic acid (GalA)

The galacturonic acid of ME kinnow pectin and commercial citrus pectin was estimated according to the method of Rodsamran and Sothornvit (2019) with few modifications. The sample was prepared for estimation by using 12 mL of concentrated H_2SO_4 diluted in 200 mL of distilled water, to dissolve 20 mg of pectin. The solution was allowed to stand for 20 min at 80 °C temperature and then refrigerated at 4 °C. 50 µl of pectin solution was syringe filtered (0.45 µm, Cole palmer, USA) and diluted with 950 µl of ultrapure water. The GalA content was determined using Hi-Plex column (Agilent Hi-Plex H, 7.7 × 300 mm, 8 µm, USA), mobile phase 5 mM H_2SO_4 with a flow rate of 0.6 mL/min in HPLC (Agilent 1290 Infinity, USA) using a standard polygalacturonic acid solution (50 mg/20 mL).

2.5. Color analysis

The colour measurements of ME kinnow pectin and commercial citrus pectin were compared using Lovibond Tintometer (Amesbury, United Kingdom) and performed in triplicates. The instrument was first calibrated by closing the white reference slider over the optics. After calibration, the cell was filled $^2/_3$ with dry powdered pectin sample and placed in SV 100 holder and covered with cap before recording color values. The values for L*, a*, b*, C*, h° and ΔE average were recorded using illuminant D65 at 10° observer angle (Khamsucharit et al., 2018).

2.6. Rheological properties

For rheological analysis, the ME kinnow pectin and commercial citrus pectin were prepared at 5 %, 10 % and 20 % (w/v) concentration and kept on heating magnetic stirrer for 4 h at 300 rpm and 50 °C for proper mixing of the samples (Nancy Picot-Allain et al., 2022). Samples were analysed by rheometer (TA Instruments, Discover HR-2, USA) at 25 °C and shear rate vs viscosity was determined. The flow behaviour of ME kinnow pectin was determined using the power law model using the given formula (5) from Ghoshal and Negi (2020).

$$\tau = K\gamma n \quad (5)$$

where τ (Pa) is shear stress, K (Pa. s^{-1}) is consistency index, γ (s^{-1}) is shear rate and n is fluid behaviour index. For Newtonian fluid n = 1, for shear thinning fluid n is $0 < n < 1$ and for shear thickening $n > 1$. ln τ vs. ln γ plot shows the fluid property of microwave extracted pectin.

2.7. Functional properties of pectin

2.7.1. Water-holding capacity (WHC)

The WHC was estimated by following the method of Hosseini et al. (2019) with slight modifications. Powdered ME kinnow pectin was mixed with ultrapure water (1:20 w/v) in centrifuge tube and vortexed vigorously, followed by centrifugation at 3500 rpm for 30 min. The supernatant was removed and weight of the residue was noted.

$$WHC = \frac{Weight\ of\ water\ retained\ (g)}{gram\ of\ pectin\ (g)} \quad (6)$$

2.7.2. Oil-holding capacity (OHC)

The OHC was determined using the method described by Hosseini et al. (2019) with minor modifications. 1 g of pectin was mixed with 10 mL of safflower oil (1:10, w/v) in centrifuge tubes and vortexed strongly, followed by centrifugation at 3500 rpm for 20 min. The

supernatant was removed and weight of the residue was observed.

$$OHC = \frac{Weight\ of\ oil\ retained\ (g)}{gram\ of\ pectin\ (g)} \quad (7)$$

2.7.3. Water-swelling capacity (WSC)

WSC was determined using method described by the previous report (Huang et al., 2021). 0.2 g of dry pectin was gradually mixed with 10 mL of ultrapure water in a measuring cylinder and covered with a stopper. The mixture was hydrated and left undisturbed at room temperature for 24 h. The volume of solid and the final volume of the hydrated pectin samples were recorded.

$$WSC = \frac{mililitres}{gram\ of\ pectin\ (g)} \quad (8)$$

2.8. Morphology, characterization, crystallinity and thermal stability

2.8.1. Fourier Transform Infrared Spectroscopy (FT-IR)

The ATR-FTIR of ME kinnow pectin and commercial citrus pectin was obtained in the absorbance mode between 4000 and 400 cm^{-1}. The powdered samples were scanned using a Fourier-Transform Infrared Spectroscopy (FTIR) coupled with ATR analysis (Agilent model: Cary 660 Series) with 4000–600 cm^{-1} scale and a variation of 4 cm^{-1}. A blank ATR cell was utilized to measure the background of the samples and the absorbance strength of each spectrum was observed.

2.8.2. Scanning electron microscopy (SEM)

For scanning, 1 mg sample of ME kinnow pectin and commercial citrus pectin was applied to black carbon adhesive tape. In order to highlight the structure, the tape fixed over the grid was exposed to gold sputtering for one minute. The structure was then visible under SEM with a 10 kV (Nikon, JEOL6000 Benchtop, Japan) at a magnification of 1000× (Wathoni et al., 2019).

2.8.3. X-ray diffraction (XRD)

The XRD (Rigaku, SmartLab SE, Japan) measurements were conducted at a temperature of 25 °C within the 10 to 80° (2θ) range. The experimental setup employed a tube current of 40 mA, tube voltage of 40 kV, a step size of 0.02 degrees per second and scanning speed 15 degrees per minute (Sucheta et al., 2020).

2.8.4. Thermogravimetric analysis (TGA)

TGA of extracted ME kinnow pectin and commercial citrus pectin samples were carried out using Perkin Elmer, STA 8000, USA to compare the thermal stabilities. A platinum crucible containing around 1 g of the sample was heated at a rate of 15 °C/minute to a temperature range of 30–800 °C. The findings were reported as weight loss percentage with increase in temperature (Kozioł et al., 2022).

2.9. Life cycle assessment (LCA) of developed process

LCA of this process was conducted on SimaPro (9.4.0) and ReCiPe 2016 Midpoint (H) V1.04/World (2010) H software which follows ISO 14040 guidelines. Cradle-to-grave approach was applied for this study and the software followed a stepwise methodology which included five main point i.e. characterization, damage assessment, normalization, weighing and addition. This study was done on functional unit of 0.1 kg of kinnow peel. The functional scale of the process design accounted for 1 kg of fresh peels per batch of primary materials. The effect of microwave-assisted pectin extraction process from kinnow peels on the environment has been determined. It is utilized for the quantitative assessment of prospective environmental impact factors involved in the product development process (Garcia-Garcia et al., 2019; Shinde et al., 2020; Nadar et al., 2022). The effect categories considered include global warming potential (GWP), stratospheric ozone depletion, ionizing radiation, ozone production, and human health. Small particle formation significantly impacts diverse ecosystems, including terrestrial, freshwater, and marine environments, by affecting organisms' toxicity, both carcinogenic and non-carcinogenic, and leading to resource depletion, such as land, minerals, fossil fuels, and water usage.

2.10. Statistical analysis

The pectin extraction was carried out in triplicates and the values were expressed as mean with standard deviation. The statistical analysis of data was done using *t*-test of two independent samples assuming equal variance with a level of confidence of 95 % using Excel Office 2019 (Microsoft, USA). Differences between the values were expressed as statistically significant at $p < 0.05$.

3. Results and discussion

3.1. Extraction of pectin using microwave heating extraction (ME)

The highest pectin production of 9.81 % was achieved for 10 min at 110 °C at 2.2 pH, with a pulse ratio of t_{on}/t_{off} = 10/10 s = 1 in ME process. It was also analysed that slight decrease of pectin yield was observed with the rise in temperature from 110–170 °C, whereas significant decrease in pectin yield at 200 °C. The study compared commercial citrus pectin with microwave-assisted extracted pectin. Both pectin has similar properties, however the ME kinnow pectin showed high galacturonic acid content and esterification. In temperature study, variation in pectin yield was observed due to use of open containers which cause evaporation that leads to deviation in pectin yield (Fig. 1). In terms of pH, the maximum yield was obtained at 2.2 pH and a significant decrease in yield of pectin was found for extraction at pH higher than 2.2. At a constant pH and temperature, pectin yield increased as extraction duration increased up to 10 min, after which it remained constant. The maximum yield for time range of 5–20 min with intervals of 5 min, was at 10 min with a pulse ratio of 10:10 i.e., t_{on}/t_{off} = 10/10 s = 1. The yield became constant after 10 min. In case of supernatant to ethanol ratio, the maximum yield was obtained at 1:4 ratio but to minimize the use of solvent and also get better yield%, 1:2 ratio was selected as reported in many prior studies (Baltazar Flores et al., 2013; Sucheta et al., 2020).

3.2. Physico-chemical characteristics of ME kinnow pectin compared with the commercial citrus pectin

The ME kinnow pectin had 833 mg equivalent weight, 7.44 % MeO, 66.67 % DE and 63.15 % GalA. It was observed from Table 1 that the extracted pectin obtained possess higher galacturonic acid (>50 %) and was of higher purity than the commercial citrus pectin. Previously, acid

Table 1
Comparison of physico-chemical composition and functional properties of commercial citrus pectin and ME kinnow pectin.

Chemical composition	Commercial citrus pectin	ME kinnow pectin
Equivalent weight (mg)	1250 ± 10.25[a]	833.33 ± 08.25[b]
Methoxyl content%	09.30 ± 00.18[a]	07.44 ± 00.19[b]
Degree of esterification%	78.95 ± 01.05[a]	66.67 ± 01.07[b]
Galacturonic acid (g/100 g)	60.63 ± 01.02[a]	63.15 ± 01.04[a]
Water holding capacity (g/g)	06.16 ± 0.096[a]	08.27 ± 0.085[b]
Oil holding capacity (g/g)	02.61 ± 0.0575[a]	03.10 ± 0.065[b]
Water swelling capacity (mL/g)	08.00 ± 1.09[a]	20.00 ± 1.25[b]

Mean ± SD (Three determinations). Different lowercase letters show statistically significant differences between the commercial citrus pectin and ME kinnow pectin sample ($p < 0.05$). The superscripts ({\,}ˆa and ˆb) indicate significant differences between the two samples for each characteristic value.

extraction using HCl yielded 14.80 % (w/w) kinnow peel pectin with degree of esterification 54.23 % (Singh & Dhillon, 2007). Ghoshal and Negi (2020) reported that 6.13 % of pectin was extracted from kinnow peel using a hot aqueous extraction technique at 90 °C for 30 min at pH 5 with equivalent weight 652.14 mg, methoxyl content 4.62 %, galacturonic acid 47.70 % and degree of esterification 37.17 %. Microwave-assisted extraction technique using organic acid in pectin yield of 16.13 %, equivalent weight 248.21 g/mol, methoxyl content 4.91 %, degree of esterification 31.36 % and anhydrouronic acid 88.93 % of citrus pectin (Mahmud et al., 2021). A reduced DE could be attributed to elevated microwave radiation levels, decreased pH, and prolonged irradiation duration and expedite the de-esterification process of polygalacturonic chains (Hosseini et al., 2016; Mahmud et al., 2021). The present findings evinced lower yield as compared to previously reported studies for citrus or kinnow pectin, however, the ME kinnow pectin was observed to be high in GalA content. Further, DE and MeO were also observed to be comparable to commercial citrus pectin.

3.3. Color analysis

Compared to the commercial citrus pectin, the extracted pectin was observed with slightly higher values of redness (+a*), yellowness (+b*) and chroma (C*) and appeared darker with lower value of lightness (L*) and hue (h^0) as shown in Supplementary Table 2. Depending on the source, commercially supplied pectin is often white, cream, or yellowish in color. A study reported color analysis utilizing a Hunter Color Lab Colorimeter for a Microwave extracted kinnow pectin where the values obtained were L* = 59.41 ± 0.17, a*= 13.63 ± 0.05 and b* = 46.42 ± 0.14 respectively (Kumari et al., 2023), whereas our study showed L* = 68.80 ± 0.98, a*= 04.50 ± 0.33 and b* =16.80 ± 0.92 for the ME kinnow pectin.

3.4. Rheological properties

Pectin's rheological behaviour is influenced by numerous factors, both intrinsic and extrinsic, including structural properties, molecular weight, concentration, pH, and co-solvents (Khubber et al., 2023; Lin et al., 2021). Both the ME kinnow pectin and commercial citrus pectin were studied for rheology at 25 °C as shear rate from 0 s^{-1} to 150 s^{-1} vs viscosity. The data as shown in Supplementary Fig. 1 evinced that with an increase in pectin concentration, the apparent viscosity of both commercial and ME kinnow pectin increased, while, an increase in shear rate led to decrease in the viscosity and fluid behaved as pseudoplastic. Shear rate (>100 s^{-1}) showed no significant change in apparent viscosity. The observed shear-thinning behaviour was due to stronger interactions among long and irregularly oriented pectin polymer chains at low shear rates. As mechanical stress increases, the cohesion between the polymer chains weaken, leading to reduced interactions. Consequently, the polysaccharide molecules form a three-dimensional network aligned with the flow direction, resulting in decreased viscosity (Nancy Picot-Allain et al., 2022). On the other hand, increasing the concentration of pectin resulted in an increase in viscosity. This might be due to higher pectin concentration which reduced the distance between molecules and enhanced intermolecular interactions such as hydrogen bonding. However, at 5 % and 10 % pectin concentrations, the intermolecular gaps may be too large for effective pectin molecule interaction, thus, viscosity did not change significantly. However, at 20 % (w/v) pectin concentration, there could be pectin chains aggregation, resulting in network formation, thus increased in viscosity was observed at higher concentration (Gawkowska et al., 2018). The impact of the concentration may be attributable to intermolecular attraction and the strength of water-solute hydrogen bonds which restrict molecular motion. Soto-Caballero et al. (2016) reported that in solutions containing large concentrations of pectin, the viscosity increases due to an increase in hydrogen bonding with the hydroxyl groups of pectin.

3.4.1. Power law model for the rheology of pectin

The power law equation was used to examine impact of viscosity on shear rate (Ghoshal & Negi, 2020). Plot of lnτ vs. ln γ was linear and n, which is also considered as slope of the plot, was observed to be less than 1 ($n < 1$), evincing the shear thinning fluid property of pectin (Supplementary Fig. 2). The power law consistency index (K) exhibited a moderate ability to forecast the overall viscosity. A greater number of relationships between pectin molecules and water leads to increased expansion of the molecules and the construction of large three-dimensional networks, which favours non-Newtonian behaviour (Soto-Caballero et al., 2016). On fitting power law, we observed that fluid behaviour (n) index of commercial pectin was higher than ME kinnow pectin. 5 % and 20 % commercial pectin had n i.e. 0.91 and 0.84, whereas ME kinnow pectin was recorded with n values, 0.84 and 0.78, respectively (Supplementary Table 1). Minimum value of n was considered to provide better mouthfeel experience (Chen et al., 2020) and results indicate that ME kinnow pectin value could serve result better mouthfeel experience as compared to commercial pectin. The relationship between consistency index (K) and fluid behaviour index (n) of both pectin samples was observed with $R^2 > 0.95$ as shown in Supplementary Table 1. Highest K value (8.31) was obtained for commercial pectin at 5 % (w/v), while lowest (2.93) was recorded for the ME kinnow pectin at 20 %. This could be due to increase in the concentration of pectin which relates with decline in the consistency index (K) as previously mentioned (Benyounes et al., 2018).

3.5. Functional properties of pectin

The functional properties of ME kinnow pectin and commercial citrus pectin are compared in Table 1. WHC, OHC and WSC values of ME kinnow pectin were 08.27 ± 0.085, 03.10 ± 0.065 and 20 ± 1.25 respectively and it was observed to be higher than commercial citrus pectin values 06.16 ± 0.096, 02.61 ± 0.0575 and 08.00 ± 1.09 for WHC, OHC and WSC respectively. These functional properties are generally influenced by the molecular weight, protein and polyphenols content, degree of acetylation and methyl esterification (Khubber et al., 2023). Polanco-Lugo et al. (2019) reported WHC (g H_2O/g pectin) of ultrasound assisted extracted pectin and conventionally extracted pectin from *Citrus reticulata* as 16.61 and 18.76, while OHC as 1.14 and 1.08, respectively. The water holding capacity of pectin influences its ability to form gel with sugar and acid. It acts as a thickening agent, stabilizer and reduces syneresis from gel or gelled products, also contributing to desired textures and improved shelf life. Additionally, it allows for reduced sugar and fat content in formulations, making pectin valuable for healthier food options. The water holding capacity of pectin is linked to its physical and chemical properties, where higher moisture content (MC) and galacturonic acid index (GAI) values, associated with more zeta potential charges, enhance water holding capacity, while a reversed correlation with equivalent weight indicates that pectin with lower equivalent weight has higher water holding capacity, suggesting its potential use in improving the quality of bakery products (Mahmoud et al., 2022). The OHC affects emulsification, stabilizing textures, and enhancing mouthfeel in various food products. Pectin's oil holding capacity contributes to the stability of emulsions in products like salad dressings, acting as a stabilizer, reduces fat content and influences texture and mouthfeel in certain formulations (Elleuch et al., 2011; Huang et al., 2021). The WSC of pectin is crucial for forming hydrogels through polymerization, influencing their structure and swelling behaviour. It plays a key role in the absorption and retention of water, affecting the overall performance of pectin-based hydrogels (Kowalski et al., 2019).

3.6. Structural characterization using FTIR, SEM, XRD and TGA

The following characteristics of ME kinnow pectin were studied and compared with the commercially extracted citrus pectin.

3.6.1. Fourier Transform Infrared Spectroscopy (FTIR)

The stretching of the O-H bond is represented by spectral region spanning from 3200 cm^{-1} to 3600 cm^{-1}. In case of commercial citrus pectin and ME kinnow pectin, the O-H stretch was observed at 3336.31 cm^{-1} and 3254.04 cm^{-1}, respectively. The spectral bands in the region of 2926 cm^{-1}–2929 cm^{-1} reflected –CH stretch and 1500 cm^{-1}–1700 cm^{-1} indicated presence of COO^{-} groups and phenolic ester of pectin. The spectral peaks were observed in both the kinds of pectin as shown in Fig. 2. In case of standard, it was observed at 1603.37 cm^{-1} which was attributed to the stretching vibration of the C=O bond in the molecule. The spectra also showed absorption peaks in the range of 1300 cm^{-1}–1400 cm^{-1} which corresponds to the $–CH_2$ groups. The absorption region in the range of 1000–1022 cm^{-1} indicated CH-O-CH groups, which was observed in commercially extracted citrus pectin at 1013.00 cm^{-1}, while in ME kinnow pectin at 1015.22 cm^{-1}. The peak 1227.17 cm^{-1} might occur due to coupling of deformed vibrational group which contain hydrogen atom like HCO, COH, CCH and HCH (Chen et al., 2020). Strong peaks in the 1150–1000 cm^{-1} range were caused by a high homogalacturonan content in the pectin (Spinei & Oroian, 2022).

3.6.2. Scanning electron microscopy (SEM)

It was observed that ME kinnow pectin and commercial citrus pectin had similar morphology and not much difference was observed having compact, wrinkled and rough surface which appeared hard. The structure of kinnow peel has smooth surface, extracted pectin observed porous structure which enhanced water-holding ability of the porous pectin and is linked to the reduced hardness of the low-fat frankfurter sausage as food application (Wongkaew et al., 2020, Ghoshal & Negi, 2020; Wathoni et al., 2019). The observed surface morphology (Fig. 3) for ME kinnow pectin was due to sudden rise in temperature in the microwave treatment. Similar observations were found in mango peel pectin and pomelo peel pectin (Liew et al., 2016; Wongkaew et al., 2020). A previous study reported a diversified structure made up of a range of compact, uneven, and rough surfaces which was extracted by citric acid and these properties were observed due to large number of neutral sugars into their framework (Spinei & Oroian, 2022).

3.6.3. X-ray diffraction (XRD)

XRD spectrum of pectin reveals the crystalline or amorphous form, which indicates its solubility behaviour for food applications. The formation of networks and retention of solutes in pectin-polymer matrixes

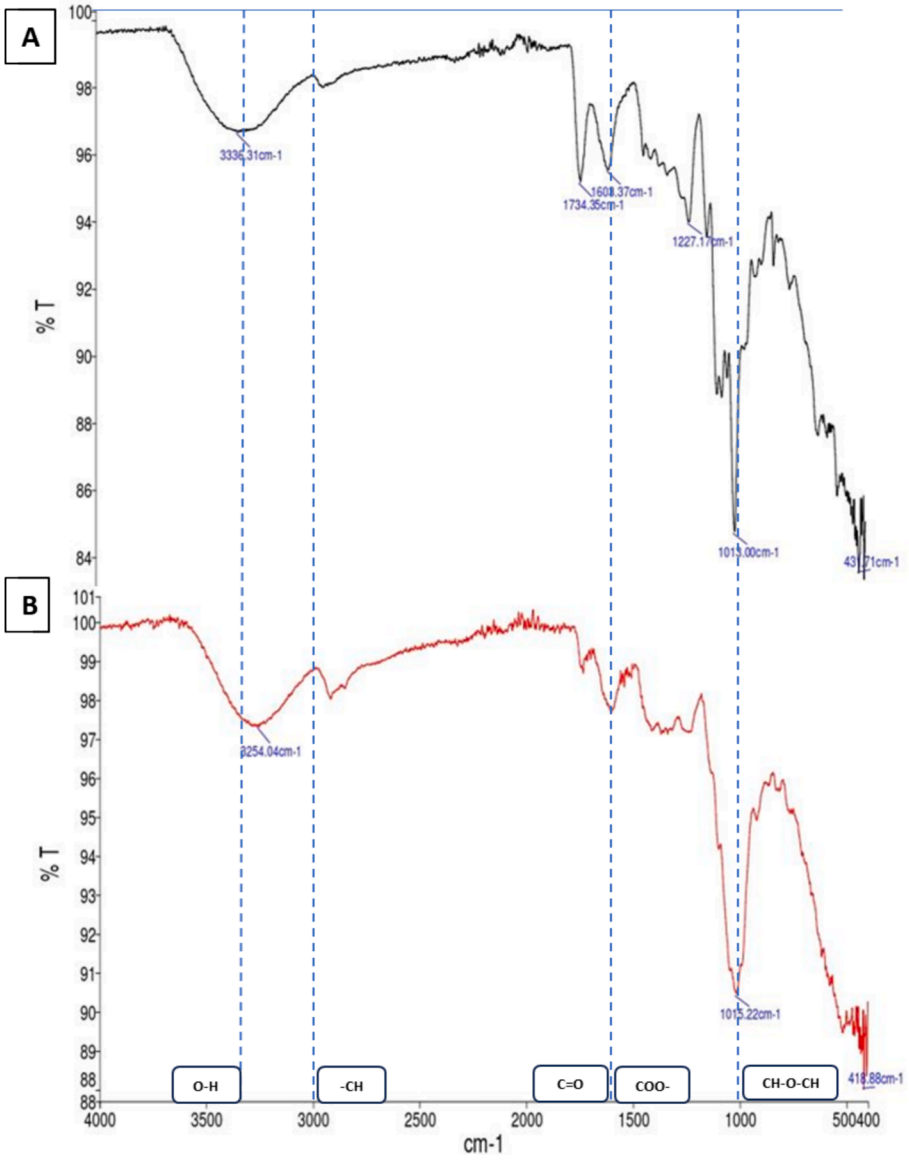

Fig. 2. FTIR spectra of a) commercial citrus pectin, b) ME kinnow pectin.

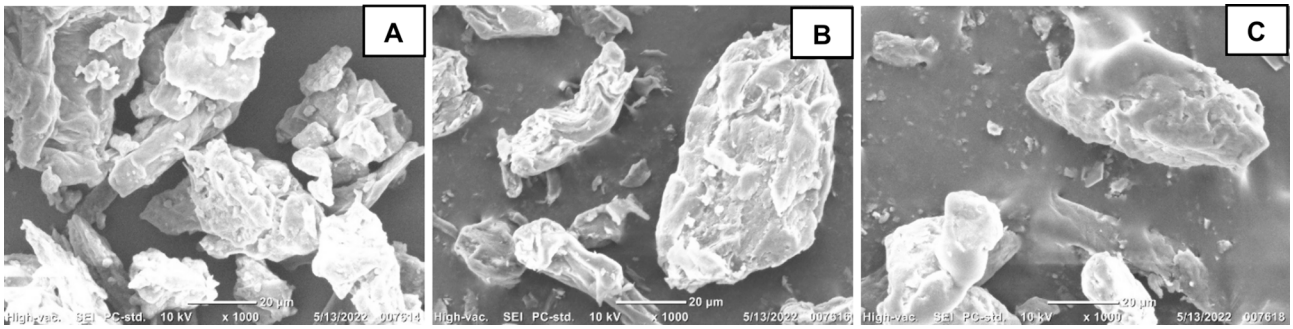

Fig. 3. SEM analysis at 1000× of a) lyophilized kinnow peel powder, b) commercial citrus pectin, c) ME kinnow pectin.

are caused by higher amorphicity. This gives viscoelasticity, which stabilizes the texture of food items. The crystalline nature of pectin samples was investigated through XRD spectra. The present study reflected that commercial pectin was more crystalline in comparison to ME kinnow pectin as shown in Fig. 4. The values of 2θ for ME kinnow pectin was observed at 14.31°, 37.93° and 43.16° and for commercial citrus pectin at 21.33° and 22. 57°. The peaks of pectin in X-ray diffraction (XRD) patterns can vary based on the source and treatment of the pectin. The XRD shows sharp peaks at 2θ value of 9°, 12.7°, 18.42°, 28.22° and 40.14°for pure pectin indicating crystalline behaviour of pectin (Kumar et al., 2010). In a study conducted by Rahmani et al. (2020), the XRD scans of sweet lemon peel pectin showed distinct peaks at various angles including 12.36°, 13.96°, 14.91°, 19.61°, 18.91°, 21.36°, 32.46°, and 36.66° (2θ), indicating the presence of both crystalline and amorphous structures in pectin. The decrease in crystallinity resulting from extraction occurs due to the hydrolysis of crystalline regions (Rahimi & Behrooz, 2011). In a study conducted by Sarkar et al. (2022), it was observed that pectin extracted from kinnow and orange exhibited higher crystallinity compared to mosambi based on the presence of narrow and sharp diffraction peaks in former two samples, whereas no distinct peak was observed in the latter sample.

3.6.4. *Thermogravimetric analysis (TGA)*

The TGA analysis of commercial and extracted pectin were compared as shown in Supplementary Fig. 3 and it was observed that in commercial citrus pectin the value of delta Y was 11.772 % at 29.8 °C, 4.893 % at 100 °C, 59.642 % at 200 °C and 18.578 % above 700 °C. In ME kinnow pectin, delta Y was observed to be 7.429 % at 29.8 °C, 8.286 % at 100 °C, 72.524 % at 200 °C and 2.718 % above 700 °C. The maximum decomposition in both the samples and standard were observed during heating at 200–700 °C. Maximum degradation occurs above 200 °C which reflect that degradation of pectin as previously reported degradation between 180 and 270 °C (Einhorn-Stoll et al., 2007). The state transition of pectin during processing, its chemical composition, and its

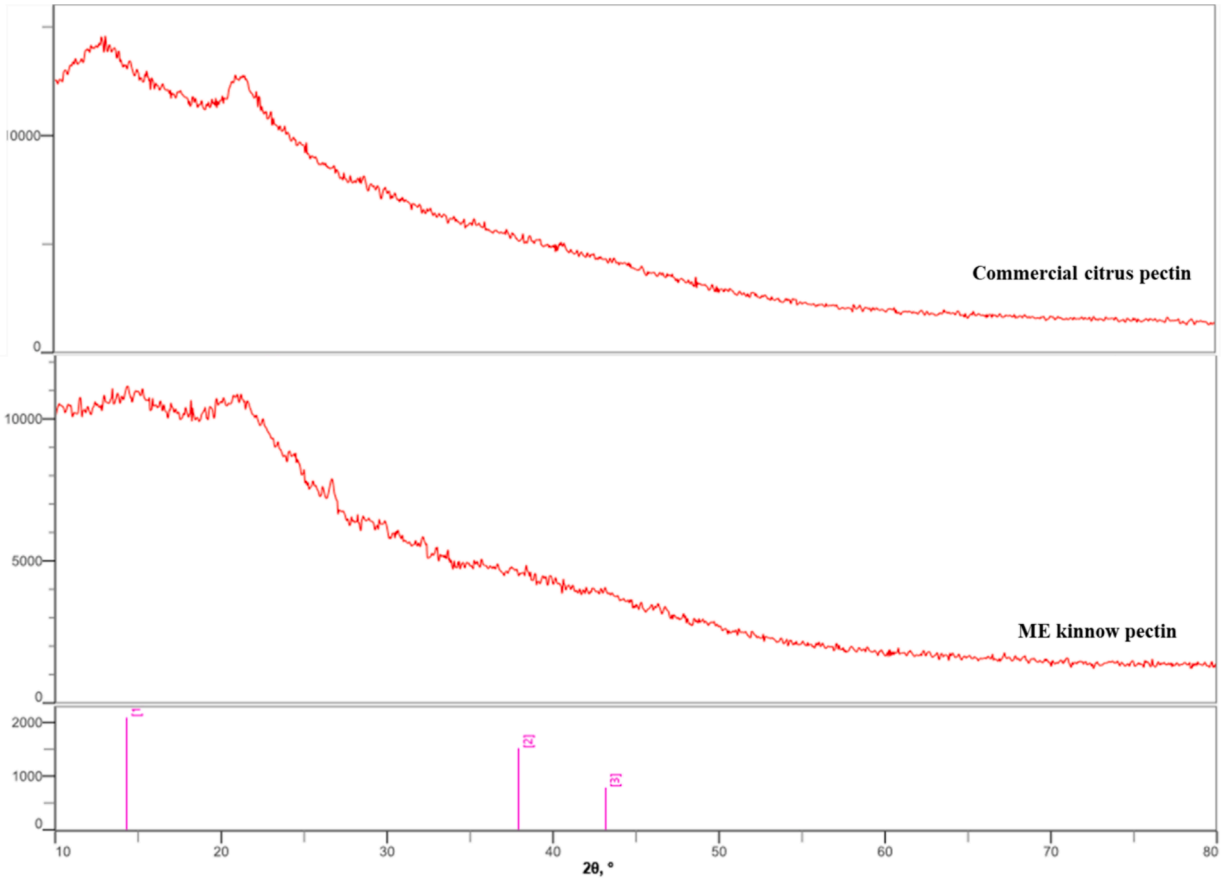

Fig. 4. XRD of a) commercial citrus pectin, b) ME kinnow pectin.

stability qualities are the three interdependent elements that determine its thermal properties, so the difference observed in commercial and extracted pectin can be any of one (Spinei & Oroian, 2022). Weight loss below 100 °C was due to evaporation of free water and pectin bound water, degradation above 100 °C due to the thermal degradation of the galacturonic acid chain, the decarboxylation of the acid side group and the carbon in the ring to generate different gaseous products and solid char and slow degradation due to solid carbon degradation due to temperature increase (Liang et al., 2022). From TGA data it was clear that pectin loss was consistent, thus this can be used for making chips, a process done at 130 °C, to reduce fat content and enhance sensory properties.

4. Life cycle assessment (LCA) of process

The main objective of conducting an LCA analysis was to evaluate how a process for development of a product can affect the environment. The environmental impacts associated with pectin production from 1 kg kinnow peel throughout its life cycle was conducted. Fig. 5 illustrates the breakdown of climate change impact at each stage of pectin production from kinnow peel. The extraction and precipitation stages, which involve the use of acetic acid and ethanol, were found to be significant contributors to climate change, as depicted in Fig. 5. The ethanol used in the precipitation and washing stages accounted for 49 % of the overall impact. This was because of ethylene hydration which is the conventional method of ethanol production that relies on a non-renewable resource. Therefore, a more sustainable alternative would be to use bioethanol derived from the fermentation of molasses obtained from cane and beet. According to Nadar et al. (2022), bioethanol from cane and beet molasses can reduce climate change impacts by 25 % and 11 % respectively. Ethanol used in this process could be used up to two cycles. The findings of this study provide valuable insights for researchers to identify critical points in the pectin production process. This knowledge can help recognize opportunities for improving the current process and implementing sustainable interventions. The LCA work need certain assumptions like, farming of kinnow and transportation were not considered but vehicles used mainly fossil fuel which contribute in CO_2 emission.

Bar diagram obtained from LCA software showed that acetic acid and ethanol contribute significantly to the extraction of pectin from kinnow peel. Acetic acid effect marine, fresh water ecosystem and little bit involve in human carcinogen whereas, ethanol get involved in marine, fresh water eutrophication, fossil fuel scarcity as well as little bit effect the terrestrial ecosystem, ozone depletion, global warming, human carcinogen and non-carcinogen. Other parameters didn't affect LCA significantly as shown in Supplementary Fig. 4.

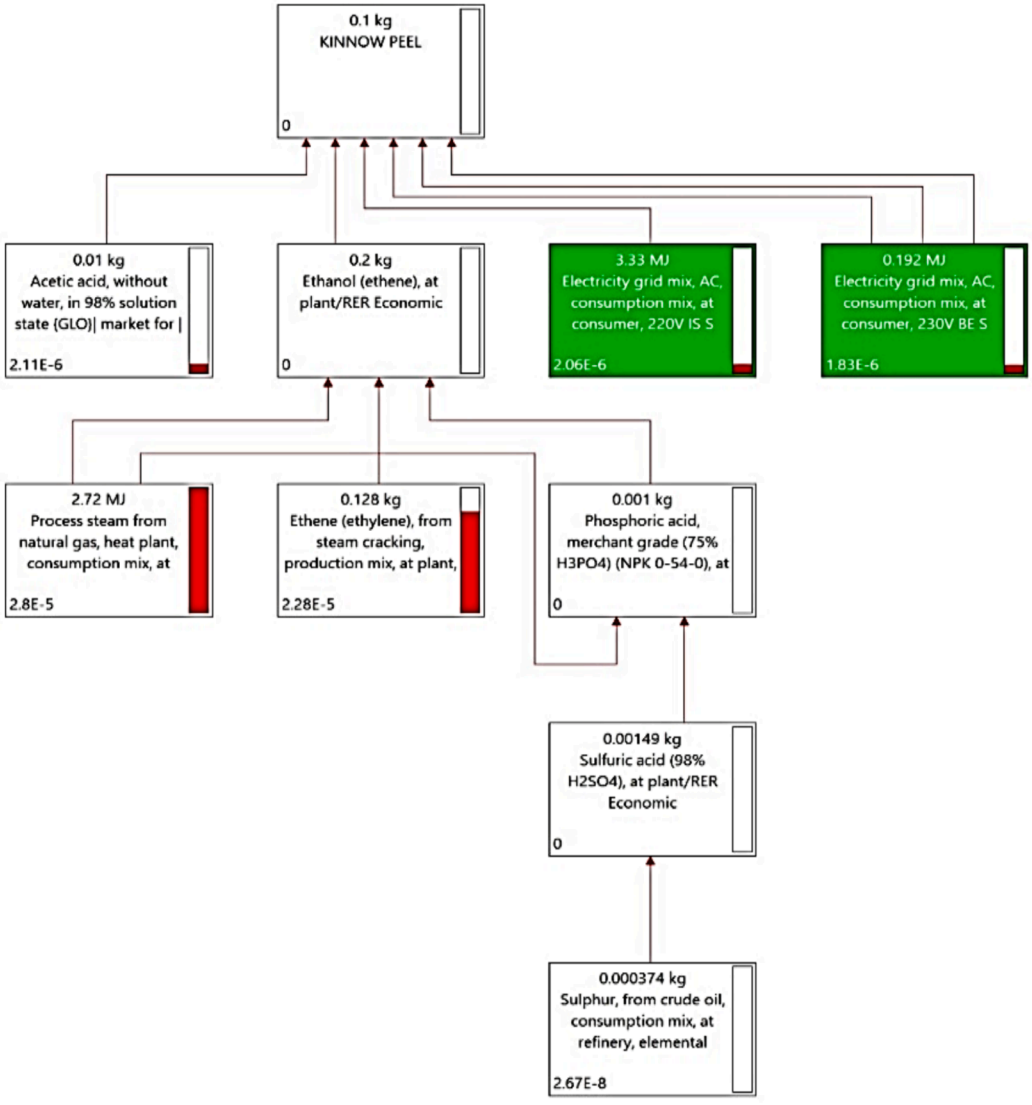

Fig. 5. Flow diagram of LCA of ME kinnow pectin.

5. Recommendation to make industry feasible process

Our study showed that acetic acid, ethanol and electricity were the main component of the process and LCA showed that ethanol and acetic acid major contributor in climate change and also in economic point of view. In order to reduce the use of ethanol crude extracted pectin was concentrated 1–3 % before precipitation, this may reduce use of ethanol up to 80 % and industries can recycle the ethanol up to 76 % via continuous distillation unit and it can be reuse which ultimately reduce cost of process as well as climate change effect (Nadar et al., 2022).

6. Conclusion

The present study analysed the effect of temperature, pH, time and ethanol concentrations in extracting pectin from dried powdered kinnow peel. The study of kinnow peel pectin-acetic acid extraction process using microwave heating, resulted in higher yield of pectin in short duration. The ME kinnow pectin showed higher purity, shear thinning behavior, and higher viscosity. ME kinnow pectin also showed better WHC, OHC and WSC as compared to commercial pectin. In LCA study, ethanol was observed to play significant role in imparting effect of pectin production process on the climate change as it is generated from non-renewable hydrated ethylene. Further, the process of obtaining valuable substances such as pectin from fruit-processing waste for waste utilization is an incredible way to reduce greenhouse gas emissions, however, the extraction must be environmentally friendly, sustainable, and cost-effective for long-term viability.

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Code availability

Not applicable.

CRediT authorship contribution statement

Muskaan Duggal: Writing – original draft, Resources, Methodology, Formal analysis, Data curation. **Devendra Pratap Singh:** Writing – original draft, Resources, Methodology, Formal analysis. **Saumya Singh:** Software, Resources, Formal analysis. **Sucheta Khubber:** Writing – review & editing, Methodology, Formal analysis. **Monika Garg:** Visualization, Supervision, Formal analysis. **Meena Krishania:** Writing – review & editing, Visualization, Supervision, Project administration, Investigation, Formal analysis, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.fochms.2024.100213.

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