

Effect of Drying on Underutilized 'Red Sitaphal' peels: Physicochemical, Structural, and Phytochemical attributes

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Abstract

Red Sitaphal is an Underutilized tropical fruit belonging to the family, Annonaceae and species *Annona squamosa*. This cultivar is known for its unique red pericarp which contributes 30–40% of the whole fruit. However, due to lesser availability and short shelf life, the Red Sitaphal peels (RSPs) remain a neglected bioresource, that can be explored for its functional and nutraceutical properties. To understand the valorization potential of the RSPs, the objective of this study was to compare three drying methods and their effect on the physicochemical, structural as well as phytochemical profile of the RSPs. Thermal drying methods, Tray and Vacuum drying and non-thermal Freeze drying were employed on the peels at different temperatures. The drying process outcomes were analyzed in terms of yield (%), moisture (%) and drying time. Further, the physicochemical (CIE-L,a,b, density, techno-functional properties, mineral content, dietary fiber), phytochemical (TPC, TFC, GC-MS profiling) and structural (FTIR) properties were determined. Due to a higher drying yield and lower moisture content, TD-45, VD-45 and FD were chosen for all the analyses. Further, higher yield of TPC and TFC in the ethanol extracts from VD and FD powders led to GC-MS profiling and a comparative evaluation of these two active RSP powder extracts. In all the parameters, the FD and VD samples performed better than TD due to absence of thermal degradation and less exposure to heat. The phytochemical compounds identified in GC-MS such as catechol, diterpenes, fatty acids etc. also suggest the diverse bioactive profile of the RSPs, which should be systematically explored in future studies. Overall, this first-of-its-kind report on Red Sitaphal peel characterization provides a baseline valorization for its application as a functional by-product.

1 Introduction

Underutilized fruits are a powerhouse of nutrients and offer wide phytochemical diversity. Meena et al. (2022) reported more than 139 species of underutilized fruits belonging to 42 families and 94 genera in India alone, among which the families such as Annonaceae, Anacardiaceae, and Myrtaceae are included. However, these species are ecologically widespread and nutritious but are not found abundantly in markets and commercial applications, due to their limited availability, short season and high perishability (Hunter et al., 2019). However, their bioactivities, including antimicrobial, anti-diabetic, and anti-Alzheimer's properties, have been highlighted, contributing to the growing research interest in them. These have a great potential for functional food development (Marappan et al., 2025).

Custard apple (*Annona squamosa* L.) is a climacteric fruit with high nutrient content and short shelf life, resulting in high postharvest losses (Sen et al., 2026). The red-pericarp variety of Custard Apple is termed as “Red Sitaphal” and considered to be one of the Underutilized fruits in India. This variety is grown in selected orchards in Maharashtra, Karnataka and Andhra Pradesh, and is also found growing in the wild as a chance seedling. Its main characteristic is the deep red to pink pigmented peel, and sweet pulp like common custard apple (Nag, Singh, & Tiwari, 2020). The edible pulp is eaten fresh, and the peel makes up a large proportion of about 35–40% of the fruit and is routinely wasted as agricultural waste. Besides economic losses, this is an environmental burden, without any valorization strategies in place to utilize them as an agro-byproduct. Red Sitaphal is notable for its unique physical features but is

underutilized and often overshadowed by other cultivars of custard apples, restricting the potential use and health benefits of this cultivar. As a lesser known variety it remains a novel bioresource, therefore the peels are discarded without any systematic characterization. Valorization of this fruit peel is still an untapped opportunity for industrial applications, especially in terms of exploring the functional and medicinal properties of the red peel which are potentially rich in anthocyanins, carotenoids and antioxidants.

As demonstrated in most agro-industrial valorization studies, such peel fractions are high in bioactive compounds, such as dietary fiber, proteins, minerals and secondary metabolites such as tannins, phenols, flavonoids. However, the processing of such by-products depends largely on suitability of valorization methods and retention of their nutritional and phytochemical integrity. The selection of the thermal processing conditions is important as different techniques such as convective tray drying and vacuum assisted drying have different impacts on the moisture content, amorphous structure and overall phenolic content of the processed powder (Redasni et al., 2026; Singh & Sharma, 2026). In particular, the antioxidant activity can be significantly reduced by thermal degradation in the process of extended drying and the surface microstructure can be changed during the dehydration process, which may affect the bioavailability of the bioactive compounds encapsulated in the shell (Dhara, Saha, Saha, & Chakraborty, 2023; Van, Le, Nguyen, Nguyen, & Nguyen, 2026).

2 Methodology

2.1 Materials

2.1.1 Chemicals and Reagents

All the chemicals, reagents and standards used in the present study were of analytical grade, and the glassware used were calibrated for accurate experimental procedures. The FCR reagent, Gallic acid, Quercetin, 99.9% Ethanol, enzymes and other reagents were procured from Sigma-Aldrich and Hi-Media, India.

2.1.2 Fruits Collection and Preparation

Physiologically mature fruits of Red Sitaphal were harvested during the month of October from the Ex-situ Biodiversity Park at University of Horticultural Sciences, Bengaluru on the basis of maturity indicators such as cracked carpels, flattened inter-areolar spaces and visible deep red colour of the peels. Fruits were immediately transported to the Food Tech Laboratory, M S Ramaiah University of Applied Sciences upon harvesting to minimize any post-harvest spoilage.

The whole fruits were thoroughly washed under tap water and surface-sanitized using a 5% NaCl-distilled water solution to remove any visible dirt or impurities. After measuring their weights, the fruits were manually separated into peels, pulp and seeds. The peel fractions were thinly sliced into uniform pieces and subsequently weighed to obtain the fresh peel weight before drying treatments.

2.2 Drying Process of Red Sitaphal Peels (RSPs)

There were two thermal and one non-thermal drying method applied in this study, namely: Tray Drying, Vacuum Drying and Freeze Drying. For all three drying method, 0.5kg of fresh peel were taken with three replicates under each treatment to ensure statistical reproducibility of the process parameters.

Tray Drying: The peel slices were spread on stainless steel trays and kept inside a Tray Dryer on separate trays for uniform drying. Three temperature settings were used for tray drying: 45°C, 50°C and 55°C. Drying was continued till constant weight of the peels were obtained after approx. 20 hours.

Vacuum Drying: A vacuum-oven dryer with a sealed vacuum chamber was used at constant pressure and temperature of 45°C, 50°C and 55°C, respectively. Thinly sliced peels were spread evenly inside the chamber and subjected to drying for approx. 19 hours till constant weight of the peels were obtained.

Freeze Drying: The peel samples were frozen initially for 12 hours at -20°C prior to freeze drying. After this, the frozen peels were immediately transferred to a Batch-type Freeze dryer where the condenser was set to -40°C at a pressure < 20 Pa for 30 hours till peels achieved a constant weight.

Post drying, the weight of the RSPs (dry basis) from each drying replicate were measured. Then, the dried peel samples were ground into a powder using a laboratory blender and the resultant powders were passed through a fine mesh sieve to maintain uniform pore size. The RSP powders were packed into air-tight aluminum pouches with a laminated layer for better storage stability. The samples were stored at 4°C and used for further analyses.

2.3 Drying Yield and Moisture Analysis

After each drying process, the drying yield was calculated on the basis of the initial fresh peel weight and final dried peel weight of the RSPs. The drying yield (%) was given as (Yan et al., 2026):

$$\text{Yield (\%)} = (\text{Final dried weight of peel} / \text{Initial fresh weight of peel}) \times 100$$

Moisture content of each dried RSPs was determined with an infrared moisture analyzer, where approximately 2 g of the finely ground powder was heated until a constant weight was achieved (Jakkranhwat & Kunchansombat, 2021). This represented the residual moisture content of the peel powders on a dry weight basis.

The drying yield and moisture content were utilized as crucial process indicators to determine the quality of RSP powders obtained via all three drying methods.

2.4 Physicochemical Properties of the RSP powders

2.4.1 Color Parameters

A handheld instrumental color analyzer (Lovibond) was used to quantify the CIE L*, a*, and b* color coordinates, where L*, a*, and b* represents brightness/lightness, red-green and yellow-blue axis in the colorimetric space (Ogrodowska, Tańska, & Brandt, 2017). The Total color difference (ΔE) from reference standard (D65 reference point) was calculated using the formula (Gordillo et al., 2018):

$\Delta E^* = \sqrt{((L^* - L_0^*)^2 + (a^* - a_0^*)^2 + (b^* - b_0^*)^2)}$; where the subscript zero denotes the values of the standard references

2.4.2 Density analysis of dried RSPs

Bulk density and tapped density of the RSP powders, Carr's index and Hausner ratio were calculated to get the characteristic powder properties and flowability. To determine bulk density, a known weight of the RSP peel powder was taken into a graduated cylinder and the initial untapped volume was measured. However, the tapped density of the samples was obtained by mechanically tapping the measuring cylinder until the volume is constant. Bulk and tapped densities were determined as the ratio of mass of the powder to the untapped and tapped volume of the powder, respectively (Šeregeli et al., 2020).

Based on the bulk and tapped density values, Carr index and Hausner ratio were calculated by standard mathematical formula, and Carr index is the compressibility of powder, Hausner ratio is the inter-particle friction and flow potential of powder (Quan et al., 2024). The calculation is given by (Klongdee, Katekhong, Jittanit, Matsukawa, & Klinkesorn, 2025):

1. Carr index = $[(\text{Tapped density} - \text{Bulk density}) / \text{Tapped density}] \times 100$
2. The ratio of Tapped density to Bulk density is known as Hausner ratio.

2.4.3 Techno-functional analysis

Techno-functional properties: water binding capacity g/g (WBC), water holding capacity g/g (WHC), oil holding capacity g/g (OHC) and solubility (%) of the RSP powders were determined according to standard method of analysis (Papoutsis et al., 2017; Rao, Poonia, & Dhewa, 2025).

2.4.4 Total Dietary Fiber (TDF)

The Dietary Fiber (%) represents the sum total of insoluble and soluble dietary fiber of a sample. TDF of RSP powders was determined as per the standard enzyme-gravimetric method by AOAC (Molina et al., 2023).

2.5 Mineral Estimation

The RSP powders were subjected to a mineral analysis using Atomic Absorption Spectrometry (AAS) as per AOAC method to determine the calcium, iron and magnesium composition in the samples (Alazb, El-Sahy, M., Sulieman, & Youssif, 2021).

2.6 FTIR Spectroscopy

The RSP powders obtained via different drying were each subjected to Fourier Transform Infrared (FT-IR) spectroscopic analysis on the basis of the structural morphology of the dried peels. K-Br pellet method as per standard protocols (Chua, Tang, Ali, & Chow, 2020) was followed, and the functional groups were detected within the range of 4000 – 400 cm^{-1} in wavelength.

2.7 Phytochemical extraction for bioactive properties

Based on drying performance and overall powder properties, TD-45, VD-45 and FD samples were subjected to conventional alcoholic extraction to prepare RSP powder extracts for phytochemical analysis.

80% ethanol was used as the preferred solvent (Anbessa et al., 2024) and the powder samples were extracted via conventional stirred maceration (Hot Plate Magnetic Stirrer, Thermofisher Scientific) in 1:20 solid to solvent ratio, for 1 hour at 40°C under dark conditions. Once the extraction was complete, the extracts were double filtered via Whatmann 40, and the filtrates were concentrated under reduced pressure (RotaVap, Buchi) at 40°C till a semi-solid RSP extract is obtained. These extracts were then used for subsequent analyses.

2.7.1 Total Phenol and Total Flavonoid Content

Estimation of Total Phenol Content (TPC) by FCR method is a standard quantification of phenolics in any samples (Boonthatui, Chongsuwat, & Kittisakulnam, 2021). For each of the prepared RSP powder extracts (TD-45, VD-45 and FD), an aliquot was taken, diluted and vortexed prior to the incubation period. A standard gallic acid curve was calibrated and the TPC was expressed in milligrams of Gallic acid equivalent (GAE) per 100 grams of the RSP powder (dry basis).

Total Flavonoid Content (TFC) corresponds to the concentration of flavonoids in the extracts. TFC was determined by the Aluminium chloride colorimetric method (Ballesteros-Vivas et al., 2019), using a standard calibrated curve of Quercetin. The values were reported as milligrams of Quercetin equivalent (QE) per 100 grams of the RSP powders (dry basis).

2.7.2 Gas Chromatography-Mass Spectrometry (GC-MS) of RSP powder extracts

The most active extracts, VD-45 and FD i.e. the vacuum dried and freeze dried peel powders were assessed for their comprehensive phytochemical profile using GC-MS analysis. A standard operating procedure was followed for the analysis with certain modifications (Hanafy, El-Shafea, Saleh, & Fathy, 2021). The extracts were reconstituted in 99.9% ethanol before analysis and a stock solution (10 mg/mL) was prepared. The solution was filtered out to eliminate any particulate matter and was then injected into the GC vials. A GC-2030 Gas Chromatograph (Shimadzu, Japan) with a DB-5MS fused silica capillary column (30 m $\times$ 0.25 mm i.d., 0.25 μm film thickness) and coupled with a quadrupole mass spectrometer was used. A constant flow rate of 1.0 mL/min of helium was used as the carrier gas, and the injector temperature was maintained at 250°C with split injection mode (split ratio 1:20). 1 μL of

the prepared extract was injected. The oven temperature was initially maintained at 60°C for 2 min, ramp 10°C/min to 280°C and maintained for 10 min for a total running time of ~ 30 min. The electron impact (EI) mode of the mass spectrometer was used with a scan range of m/z 40–600 and an ion source temperature of 200°C and an interface temperature of 250°C. Tentative assignments were made based on the identification of peaks with those found in the NIST library V.26, and published mass spectra. After the chromatograms were generated with detected compounds, the relative abundance of the identified secondary metabolites was determined by peak area normalization to estimate the qualitative composition of the extracts (Aboubi et al., 2022).

2.8 Statistical analysis and Data processing

In the present study, all the experimental results were statistically analyzed using Jamovi (v2.3) for One-way ANOVA, and individual Data were shown using Mean±Standard deviation (SD) with values measured in triplicates (n = 3). The statistical significance was considered at p < 0.05.

3 Results

3.1 Drying properties of RSPs

Table 1 Tray Drying, Vacuum Drying and Freeze Drying of RSPs and their respective drying time, yield, and moisture content across different temperatures				
Drying method	Temperature	Yield (%)	Moisture (%)	Drying time (h)
TD	45°C	33.48 ± 2.5 ^a	6.87 ± 0.04 ^b	20.9 ± 0.59 ^e
TD	50°C	31.38 ± 1.23 ^g	5.39 ± 0.04 ^f	19.31 ± 0.17 ^b
TD	55°C	27.52 ± 1.48 ^c	5.27 ± 0.06 ^d	18.2 ± 0.14 ^e
VD	45°C	27.95 ± 1.98 ^b	5.85 ± 0.09 ^g	19.22 ± 0.25 ^c
VD	50°C	25.99 ± 0.4 ^d	4.9 ± 0.05 ^a	18.47 ± 0.05 ^g
VD	55°C	22.73 ± 0.9 ^e	4.25 ± 0.04 ^c	17.93 ± 0.09 ^a
FD	-40°C	29.05 ± 3.95 ^f	4.83 ± 0.06 ^e	30.22 ± 0.21 ^f

TD: tray drying, VD: vacuum drying, FD: freeze drying. Data presented as Mean ± SD with triplicate values for each drying method; superscript letters (a,b,c,d,e,f,g) within columns indicate statistically significant differences between drying treatments at p < 0.05.

The yield of the thermal treatments decreased with increasing drying temperature and was maximum for tray drying at 45°C (33.48%) and minimum at 55°C (27.35%), suggesting the occurrence of higher losses of solids and/or structural degradation at higher temperatures. The VD and TD samples had a lower yield

at the same temperatures, while the yield of FD at -40°C was in between of the two others. This corresponds to similar studies in literature, which have shown the preservation of structural integrity in freeze drying of fruit peels with higher sublimation losses depending on the porosity of matrix (Ong, Harith, Shahril, Shahidan, & Hapidin, 2021). The moisture contents of the samples decreased with increasing temperature for both tray drying and vacuum drying, and the moisture of VD-55 (4.25%) sample was minimum, followed closely by VD-50 (4.90%) and freeze-dried sample (4.83%) thus showing that the VD and FD are effective to achieve safe storage moisture content. While slightly higher moisture (6.87%) was maintained by TD-45 than other treatments, this moisture level is still acceptable for dried peel applications and is similar to citrus and avocado peels moistures reported (approx. $50\text{--}55^{\circ}\text{C}$ (Ramírez-Aguirre et al., 2026). The drying time for both thermal drying techniques reduced with the increase in drying temperature from 20.9 h to 18.2 h for TD and 19.22 h to 17.93 h for VD, while FD was the highest time-consuming method (30.22 h). Such drying time patters are consistently seen in other fruit peel drying studies, where FD retains structural and functional integrity but remains the least energy-efficient method (Yang et al., 2024). This study also shows that lower drying temperatures (both TD and VD at 45°C) yield more peel, but take slightly longer time, while freeze drying at -40°C yields moderate peels but with very low moisture content, and takes much longer drying time. In summary, TD-45 and VD-45 are balanced thermal treatments and demonstrates suitability to be analyzed further.

For the three drying techniques, the total time required for freeze drying was much longer than for convective drying, possibly because of the need for phase change involved in sublimation in vacuum (Répás, Nagy, Polgár, & Győri, 2024). On the other hand, the convective drying methods enabled faster moisture removal, but the moisture extracted decreased as a result of the more severe thermal exposure (Quispe-Fuentes, Poblete, Aranda, & Vega-Gálvez, 2023). The higher yields of the tray dried samples at 45°C indicates that low-intensity thermal treatments can maintain the structural integrity and prevent degradation of bioactive compounds to a greater extent than other convective processes.

All the treatments were effective to inhibit microbial proliferation and to prevent the deterioration of the shelf-life because moisture content in all drying conditions was significantly lower than 7% (Kaveh et al., 2025). Moreover, comparative study of these drying parameters shows that tray drying has better process efficiency, but the drying in vacuum and freeze conditions are better in maintaining the initial physicochemical matrix of *Annona squamosa* peels, which depends on moisture. The findings are in line with earlier studies that showed that vacuum-based processing would be more effective at preserving bioactive markers than conventional tray-drying processes (Ramachandran, Nadimi, Cenkowski, & Paliwal, 2024).

3.2 Drying performance and sample selection

TD-45 and VD-45 were selected as the representative thermal drying treatments for further characterization alongside the FD treatment as the non-thermal method. These two drying treatments among the thermal methods i.e. tray drying and vacuum oven drying, provided a favorable performance between product yield, final moisture content, and drying time. Moreover, the selection on the basis of combined assessment of drying parameters facilitates a direct comparison of quality attributes at the

same drying temperature. This approach ensures that the subsequent analysis of physicochemical and phytochemical properties remains grounded in the optimal process conditions identified for each drying mechanism (Redasni et al., 2026).

3.3 Physicochemical Characterization

3.3.1 Color Parameters

As per the instrumental colour analysis of RSP powders (Table 2), FD had the highest lightness ($L^* = 59.86$) and yellowness ($b^* = 13.51$) and was intermediate in redness ($a^* = 8.62$) while TD peel at 45°C had the lowest lightness ($L^* = 45.21$) and yellowness ($b^* = 9.14$) and moderate redness ($a^* = 8.20$). VD at 45°C had intermediate lightness ($L^* = 54.45$) but the highest a^* ($a^* = 9.77$), and b^* ($b^* = 11.28$) was between TD-45 and FD. The total color difference (ΔE) was the highest for TD-45 (56.15), followed by VD-45 (47.93) and the lowest for FD (43.25), which indicates FD having minimal difference in L^* , a^* , b^* values while the maximum difference was observed in L^* , a^* , b^* values for TD-45.

Table 2
CIE(Lab) values of RSP powders across different drying methods

Drying method	L^*	a^*	b^*	ΔE
TD-45	45.21 ± 0.02^A	8.2 ± 0.03^B	9.14 ± 0.04^C	56.15 ± 0.04^A
VD-45	54.45 ± 0.30^C	9.77 ± 0.06^A	11.28 ± 0.07^B	47.93 ± 0.29^C
FD	59.86 ± 0.15^B	8.62 ± 0.07^C	13.51 ± 0.04^A	43.25 ± 0.13^B

TD: tray drying, VD: vacuum drying, FD: freeze drying. Data presented as Mean $\pm$ SD with triplicate values for each drying method; uppercase letters (A,B,C) in superscript within columns indicate statistically significant differences between drying treatments at $p < 0.05$.

The higher L^* value found in freeze dried samples may be due to the maintenance of structural integrity and the lesser degree of oxidative browning reaction compared to convective drying methods (Lekjing, Venkatachalam, Charoenphun, & Noonim, 2024). Considering the lower L^* values for the TD samples, it is likely that the pigment degradation and the presence of non-enzymatic browning products tend to increase with convective heating compared with vacuum heating and freeze-drying, as also seen in other fruit matrices. Similar color parameters reported in sublimation studies show that the total color change during freeze-drying remains minimal and natural pigments are retained as compared to heat intensive methods (Shuen et al., 2021).

3.3.2 RSP Powder Properties

This section shows the analysis on various parameters of RSP powders focusing on density of the powders, hydration behavior, powder flowability, and total dietary fiber. These are critical indicators of the

structural matrix and reconstitution behavior of processed peel powders (Mphahlele, Fawole, Makunga, & Opara, 2016).

The results show that bulk density of TD-45 > VD-45 > FD (Table 3) which reflects the inverse relationship of the thermal drying processes in producing powders with higher bulk densities. In case of tapped density as well, TD-45 showed a higher value (0.76 ± 0.03 g/mL) compared to VD-45 (0.54 ± 0.02 g/mL) and FD (0.42 ± 0.02 g/mL), indicating that prolonged thermal exposure can possibly increase compactness in dried powders and also lead to collapse of the matrix. Both bulk and tapped density in FD sample was found to be lower than the thermal methods, which possibly explains the lower Carr index and Hausner ratio in FD as well. These two parameters reflect the flow properties of the FD powder, which are found to be 10.61% and 1.12, thus showing that the FD powder exhibits good flowability (Rao, Poonia, & Dhewa, 2025).

Table 3
Comprehensive physicochemical properties of the Tray dried, Vacuum dried and Freeze dried RSP powders.

Drying method	Bulk density (g/cm ³)	Tapped Density (g/cm ³)	Carr Index (%)	Hausner ratio	WBC (g/g)	WHC (g/g)	OHC (g/g)	Solubility (%)	Total Dietary Fiber (%)
TD-45	0.55 ± 0.01^a	0.76 ± 0.03^b	27.91 ± 1.94^c	1.39 ± 0.04^b	5.14 ± 0.04^c	6 ± 0.08^b	2.19 ± 0.02^a	16.7 ± 0.15^c	60.56 ± 0.03^b
VD-45	0.44 ± 0.03^c	0.54 ± 0.02^a	18.97 ± 5.83^b	1.24 ± 0.09^a	5.77 ± 0.09^b	6.76 ± 0.04^a	2.41 ± 0.04^c	18.53 ± 0.12^b	58.36 ± 0.41^a
FD	0.37 ± 0.01^b	0.42 ± 0.02^c	10.61 ± 4.9^a	1.12 ± 0.06^c	6.52 ± 0.05^a	7.64 ± 0.06^c	2.58 ± 0.02^b	19.94 ± 0.23^a	57.48 ± 0.26^c

TD: tray drying, VD: vacuum drying, FD: freeze drying. Data presented as Mean ± SD with triplicate values for each drying method; lowercase superscript letters (a,b,c) within columns represent statistically significant differences between groups at p < 0.05.

The techno-functional properties of dried peel powders dictate their application, incorporation and suitability in different food matrices. In this study, freeze dried powders demonstrated a substantially superior WBC (6.52 g/g), and WHC (7.64 g/g) as compared to both tray dried and vacuum dried samples. The solubility (approx 20%) of FD powder was also significantly higher than the thermally dried RSP powders. These characteristics of the FD sample hence indicate that the porous structure is relatively retained during freeze drying as reflected by the higher solubility and hydration behavior (Júnior et al., 2023; Uscanga, Salvador, Camacho, & Martínez-Navarrete, 2021). Whereas, the poorly exhibited WBC, WHC, OHC as well as solubility of TD powder indicates much higher thermally induced collapse of the porous matrix of the dried material. However, VD powder showed moderately fair flowability and

hydration values, suggesting that the lower pressure maintained during vacuum drying partially preserves structural damage as compared to the complete densification observed in tray drying (Bas-Bellver, Barrera, Betoret, & Seguí, 2022; Rosidi, Yaacob, Yusof, & Yusof, 2021). The varying hydration properties across WBC, WHC, OHC and solubility corresponds to differences arising out of varying temperature exposures and pressure conditions within each drying method (Yan et al., 2026).

Along with the structural variations as observed by the density values and flow properties, the Total Dietary Fiber (%) of the three dried powders were also analyzed (Table 3). The TDF was found to be slightly higher in TD (60.56%), followed by VD (58.36%) and FD (57.4%). Due to the prolonged thermal exposure during tray drying, there is a possibility of increase in the concentration of insoluble components within the peel matrix, which may lead to the variation in the observed results. Overall, all three RSP powders exhibit a high TDF content which could be useful in development of fiber-rich products or in utilization as a source of fiber extraction for targeted functional applications (Lesmana et al., 2021).

3.5 Mineral Estimation

The results in this study showed varying mineral compositions (Table 4) among the three drying treatments. FD powders consistently exhibited a higher Calcium (280.02 mg/100g) and magnesium (340 mg/100g) content as compared to the thermally dried samples (TD and VD). These minerals are susceptible to heat and often degrades when subjected to prolonged high temperatures. Therefore, the reduced retention of all the minerals in TD-45 are in accordance with similar studies on fruit peel compositions (Alshallash et al., 2023).

Table 4
Mineral composition in RSP powders

Drying method	Calcium (mg/100g)	Iron (mg/100g)	Magnesium (mg/100g)
TD-45	180.58 ± 0.8 ^d	12.04 ± 0.06 ^e	230.23 ± 0.32 ^f
VD-45	160.04 ± 0.06 ^e	14.26 ± 0.23 ^f	220.07 ± 0.06 ^d
FD	280.02 ± 0.03 ^f	7.94 ± 0.12 ^d	339.63 ± 0.55 ^e

TD: tray drying, VD: vacuum drying, FD: freeze drying. Data presented as Mean ± SD with triplicate values for each drying method; lowercase letters in superscript (d,e,f) within columns represent statistically significant differences between drying methods at p < 0.05.

3.6 FTIR spectroscopic analysis

The RSP powders obtained from three drying methods, TD-45, VD-45, and FD were analyzed using FTIR spectroscopy to assess the presence and distribution of key functional groups within each dried peel matrix. The results of the FTIR spectra of TD-45 (Fig. 1), VD-45 (Fig. 2) and FD (Fig. 3) showed several characteristic absorption peaks consistent with the chemical structure of polysaccharides, proteins, and

polyphenolic compounds typically found in fruit tissues. Specifically, the broad band observed between 3200 and 3400 cm^{-1} is attributed to O-H stretching vibrations, indicating the presence of hydroxyl groups in cellulose and polyphenolic compounds (Talens, Arboleya, Castro-Giráldez, & Fito, 2016).

According to the FTIR spectra of VD-45, the broad O-H stretching band shows the most pronounced intensity at 28.24%T, indicating retention of hydroxyl-bearing compounds under vacuum environment. Similarly, the freeze-dried powder shows a deep and sharp peak in the same region, also confirming the retention of phenolic and hydroxyl-compounds. The peak stretching is relatively weak in TD-45, which indicates thermal induced degradation of compounds. Moreover, the signal intensities in the region from 1600 to 1750 cm^{-1} corresponding to carbonyl stretching vibrations of pectins and esters were sharper in FD and VD samples, indicating higher preservation of the structural integrity of these polysaccharides in these samples, compared to the flattened signals in TD-45 (Chauhan, Mohite, & Sharma, 2026).

In summary, the chemical structure of the peel matrix was better preserved as per the spectroscopic profiles of VD45 and FD, indicating prevention of unwanted thermal degradation of sensitive functional groups, such as carboxylic acids and hydroxyl groups (Khan, Sohail, Ghazanfar, Ahmad, & Riaz, 2024; Yu et al., 2024). Furthermore, below 1500 cm^{-1} , bands in the "fingerprint region" provide additional evidence for specific structural differences in the polysaccharide profiles, thus corroborating aggressive thermal drying drives bond dissociation (Pereira et al., 2023).

3.7 Phytochemical properties of RSP powders

3.7.1 Total Phenol and Total Flavonoid Content of RSP extracts

The results showed a higher phenolic and flavonoid content in the freeze-dried and vacuum-dried RSP powder extracts, as compared to the significantly lower values found in the TD-45 sample. A TPC of 149 mg GAE/g of FD powder reflects the superiority of sublimation process in preserving the bioactive compounds and phenols within the peel powders.

In contrast, the thermal denaturation of phytochemicals such as phenols and flavonoids potentially causes lower retention of such compounds in the TD-45 extracts. VD-45 showed moderate TPC of 93.5 mg GAE/g DW, and a similar TFC of 5.6 mg QE/g DW to that of the FD extract. This trend in reporting TPC and TFC values is consistent with several fruit peel extracts, where absence of oxidation and thermal stress leads to better retention of phytochemicals (Quan et al., 2024).

Table: TPC and TFC values of TD-45, VD-45 and FD extracts of RSP powders. values taken in triplicate (n = 3) and lowercase letters (a,b,c) within columns denotes significant differences between groups at $p < 0.05$

Extract	TPC (mg GAE/g DW)	TFC (mg QC/g DW)
TD-45	9.01 ± 1.94 ^a	1 ± 0.06 ^b
VD-45	93.5 ± 3.97 ^c	5.6 ± 0.06 ^a
FD	149.00 ± 2.33 ^b	6.61 ± 1.0 ^c

Based on the retention of phenol and flavonoids in the VD-45 and FD extracts, these two samples were further analyzed through GC-MS to identify specific bioactive metabolites and volatile compounds preserved by these drying techniques. The identification of these compounds reveals how lower processing temperatures in vacuum and freeze-drying prevent the thermal degradation of heat-sensitive phytochemicals, thereby maintaining superior bioactivity compared to tray drying (Ling et al., 2025).

3.7.2 GC-MS profiling of RSP extracts

A total of 50 compounds were detected in each of the VD-45 and FD extracts, as shown in the GC-MS chromatograms (Figs. 4 and 5).

For the purpose of drawing comparisons between the compounds identified in both the extracts, the GC-MS datasets were normalized on the basis of total peak area and percent-abundance of shared compounds. This comparative assessment indicates that both methods were effective at maintaining the qualitative spectrum of metabolites but compares well with previous work finding that low temperature sublimation minimized enzymatic degradation during processing and allowed greater recovery of thermally labile diterpenoids (Cao et al., 2021; Ibrahim et al., 2023).

N.B: FD: Freeze dried extract; VD: Vacuum dried extract; Dodecahydrophenanthrene carbaldehyde: (1R,4aR,4bS,7R,10aR)-1,4a,7-Trimethyl-7-vinyl-1,2,3,4,4a,4b,5,6,7,9,10,10a-dodecahydrophenanthrene-1-carbaldehyde; Cycloprop[e]azulen Derivative: 1H-Cycloprop[e]azulen-7-ol derivative; TUD: (1S,2E,6E,10R)-3,7,11,11-Tetramethylbicyclo[8.1.0]undeca-2,6-diene; Cyclohexanone: Cyclohexanone, 2-(hydroxymethyl)-; DOPA-TMS: 1-(3,4-Dihydroxyphenyl)-2-(methylamino)ethan-1-one, 2TMS

Based on the normalized abundance data, the heatmap shows distinct clusters of phenolics and terpenoid constituents, confirming that lyophilization retains a higher relative concentration of oxygen-functionalized metabolites prone to hydrolytic degradation at elevated temperatures (ElNaker et al., 2021).

4 Conclusion

This is the first systematic study on the effect of thermal and non-thermal drying methods on an underexplored 'Red Sitaphal' peel, which emphasizes on the suitability of drying method based on the physicochemical, phytochemical and structural properties of the dried peel powders. Thus, this research

establishes a valorization pathway for functional applications of this agricultural by-product while also promoting the underutilized fruit cultivar. The comparative analysis between the drying of RSPs establish a baseline characterization of this specific cultivar, across multiple processing outcomes. Freeze Drying consistently showed better result in phytochemical retention, better structural integrity and higher physicochemical properties. However, it is an energy intensive process with limitations. Hence, Vacuum Drying emerges as a viable drying method which can moderately retain the functional properties while also being cost-effective. Future studies should focus on process optimization and in-depth validation of in-vitro biological activities of the RSP powders and extracts.

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Figures

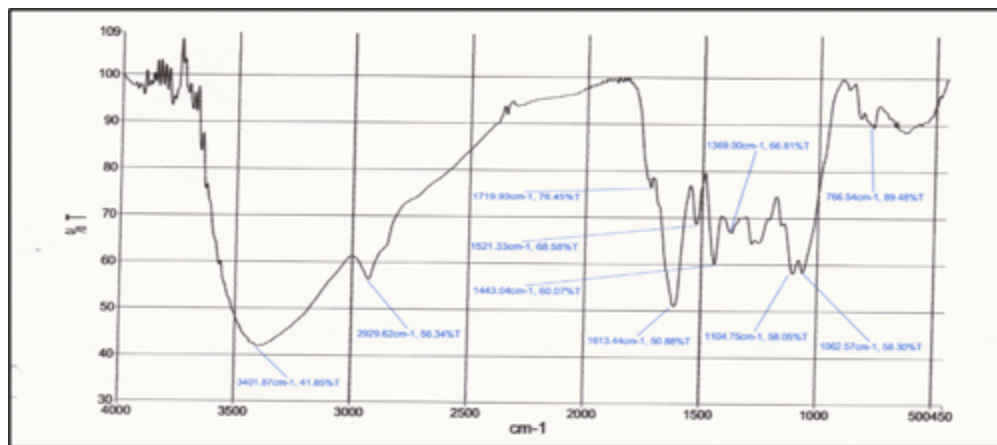


Figure 1

K-Br FTIR spectra of TD-45

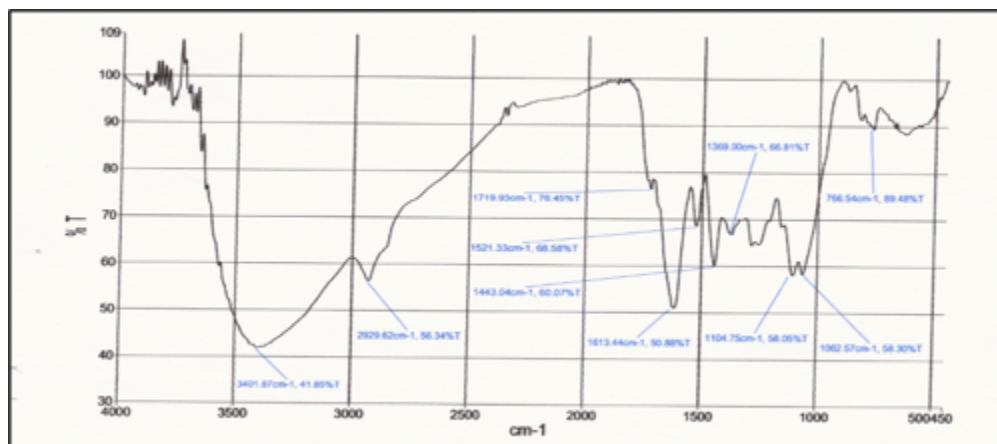


Figure 1

K-Br FTIR spectra of TD-45

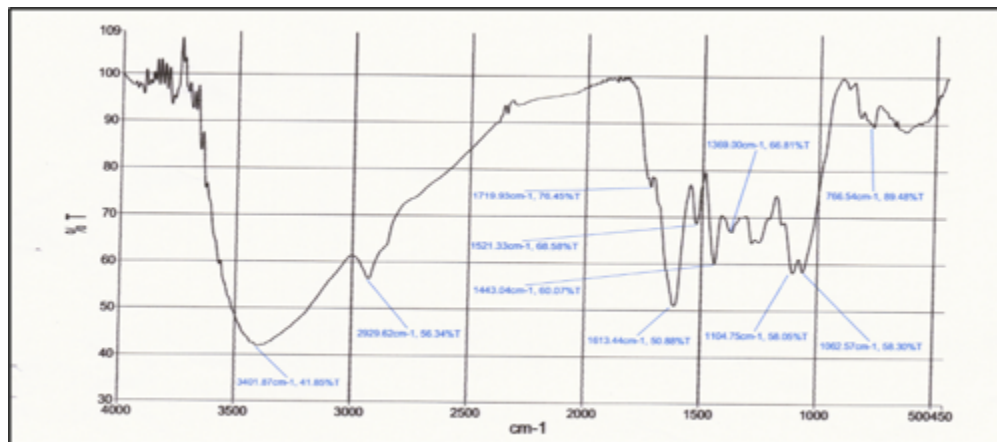


Figure 1

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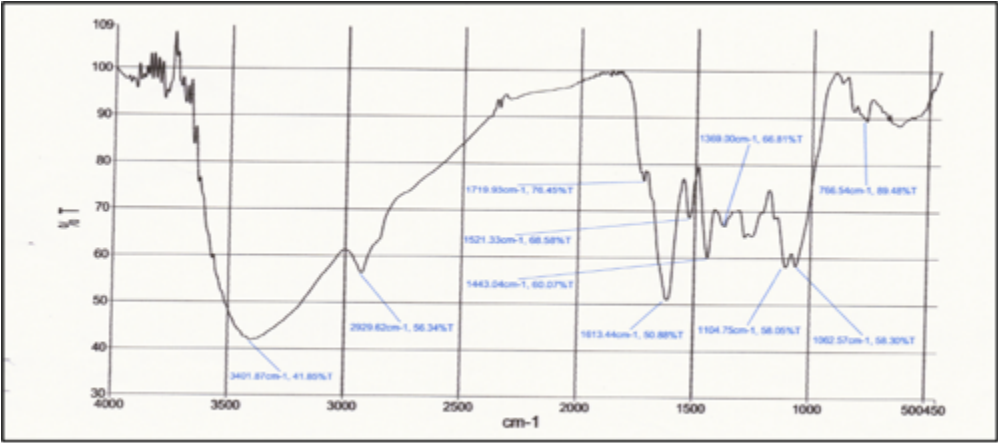


Figure 1

K-Br FTIR spectra of TD-45

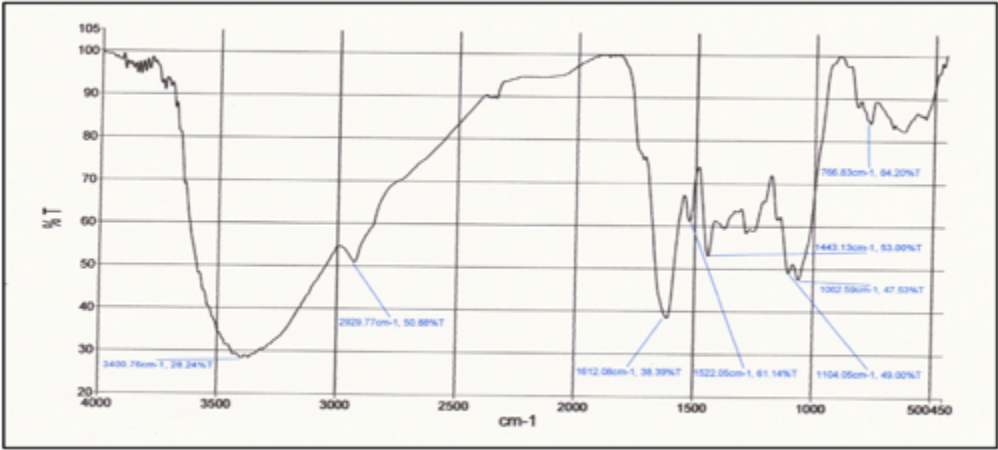


Figure 2

K-Br FTIR spectra of VD-45

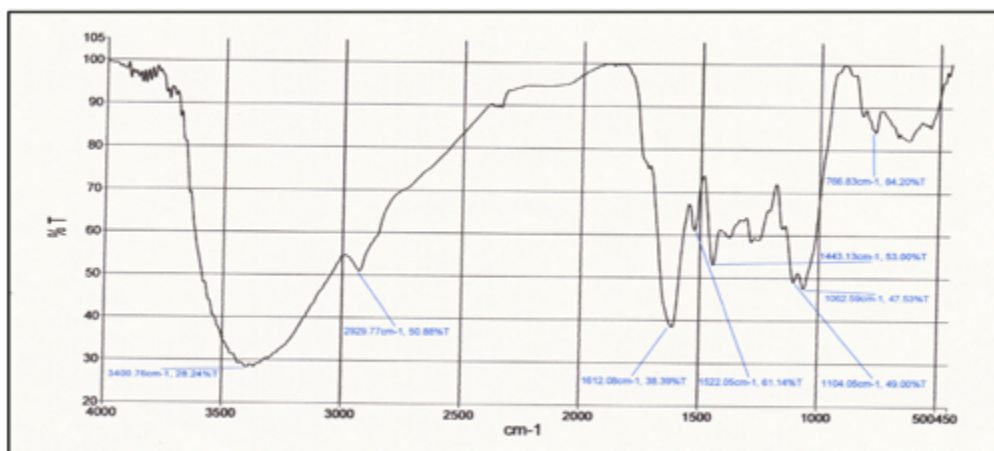


Figure 2

K-Br FTIR spectra of VD-45

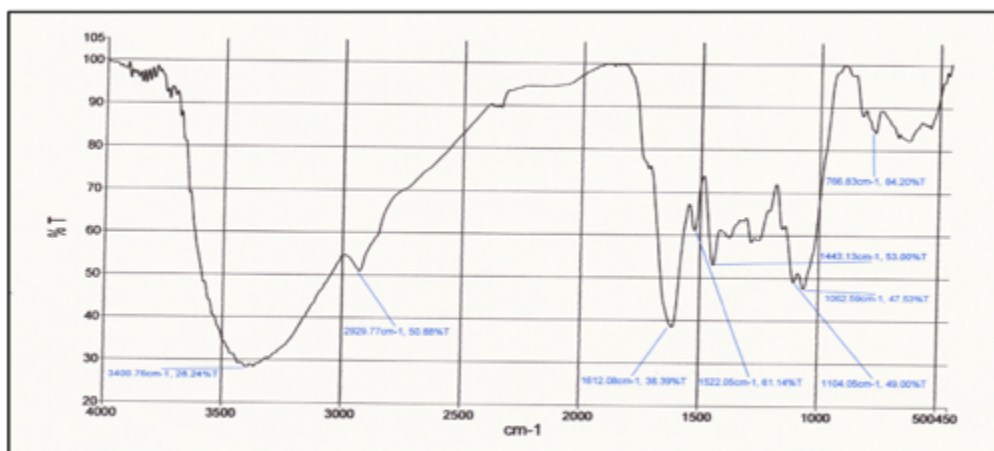


Figure 2

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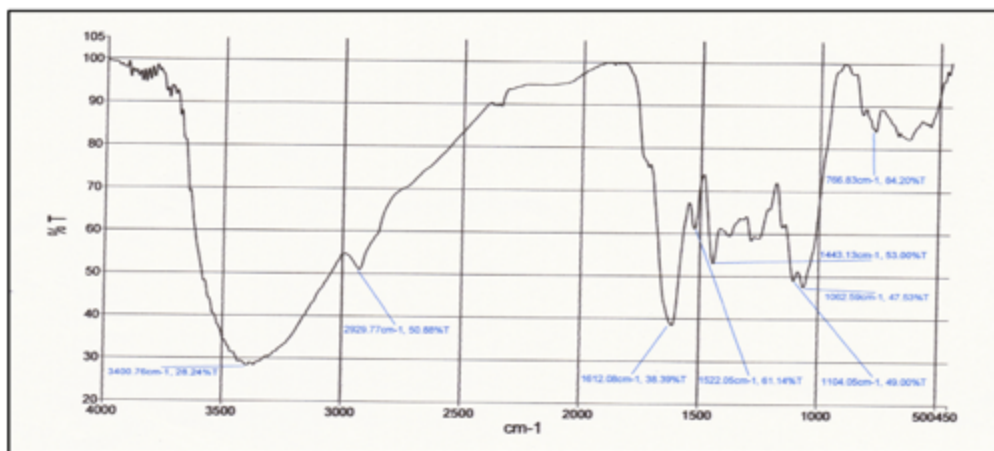


Figure 2

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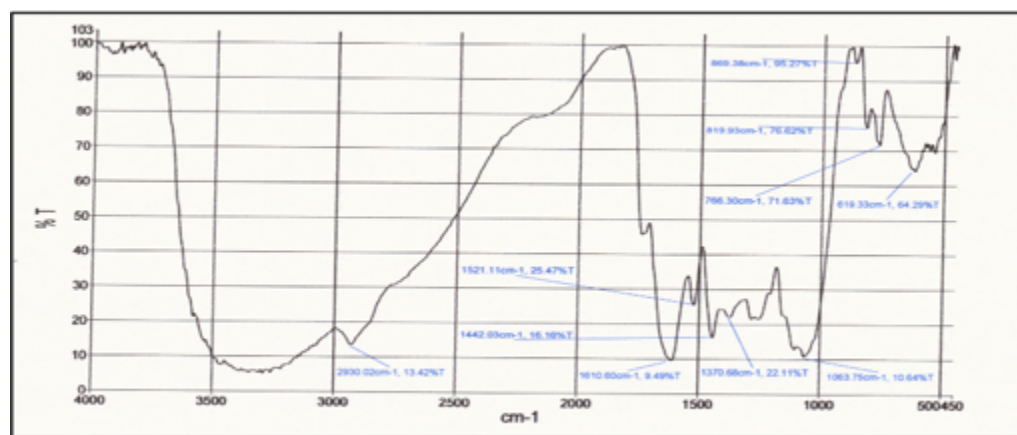
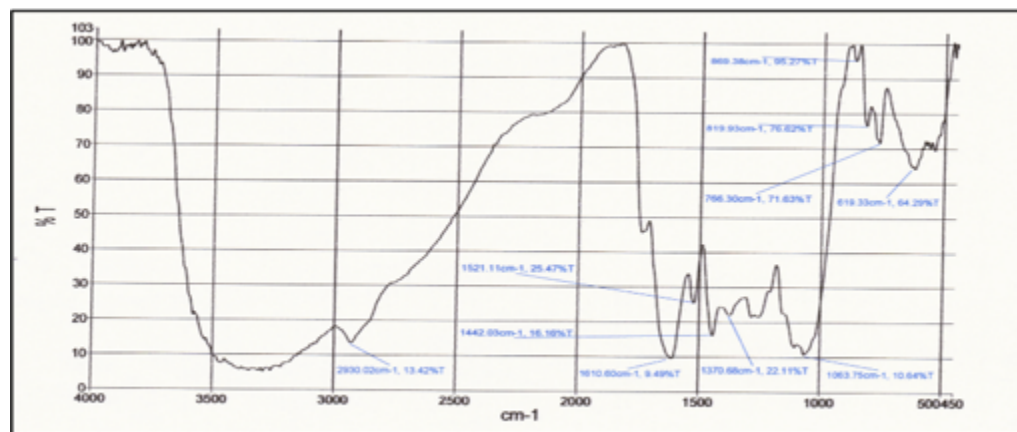
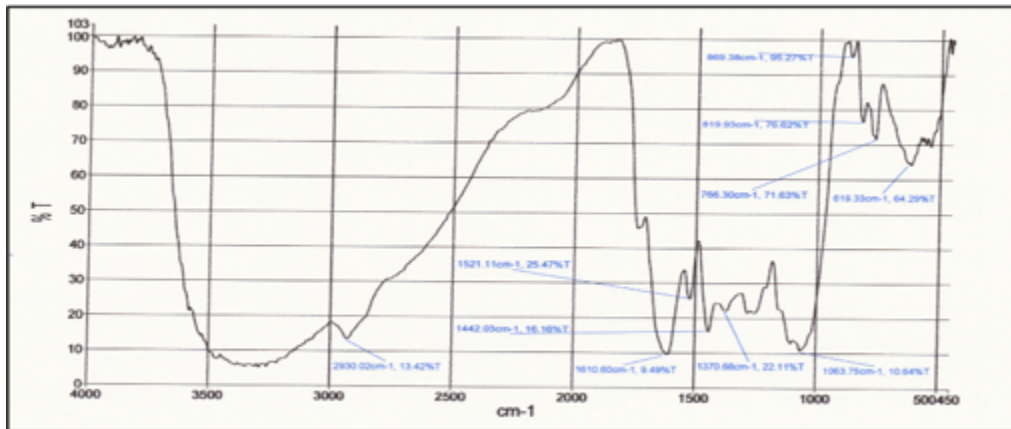


Figure 3

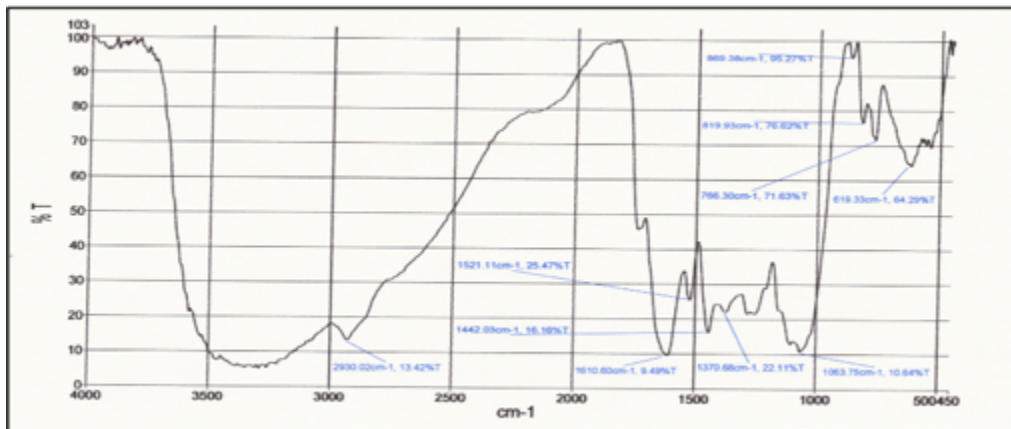
K-Br FTIR spectra of FD



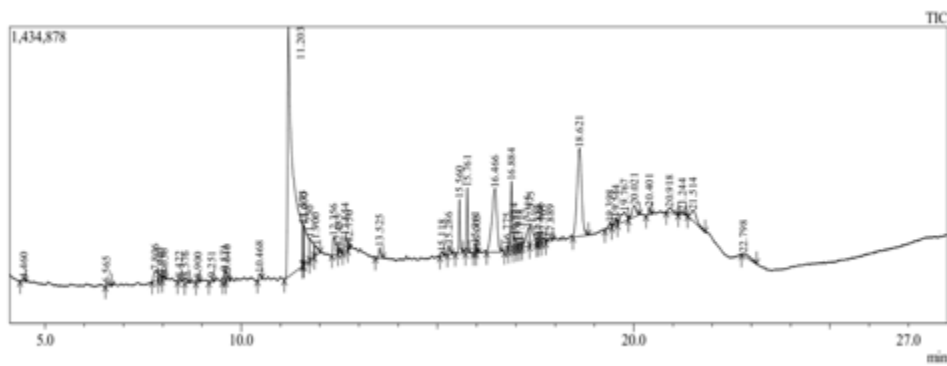
K-Br FTIR spectra of FD



K-Br FTIR spectra of FD



K-Br FTIR spectra of FD



Total ion chromatogram of ethanol extract of Freeze-dried RSP powder



Total ion chromatogram of ethanol extract of Freeze-dried RSP powder



Total ion chromatogram of ethanol extract of Freeze-dried RSP powder



Total ion chromatogram of ethanol extract of Freeze-dried RSP powder

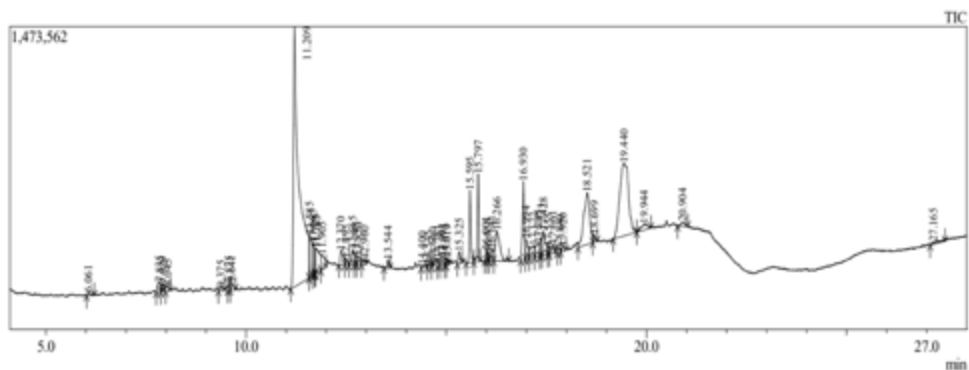


Figure 5

Total ion chromatogram of ethanol extract of Vacuum-dried RSP powder

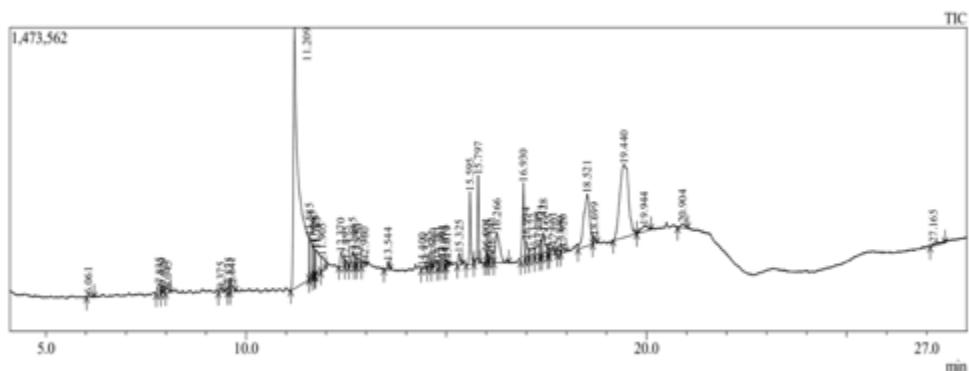


Figure 5

Total ion chromatogram of ethanol extract of Vacuum-dried RSP powder

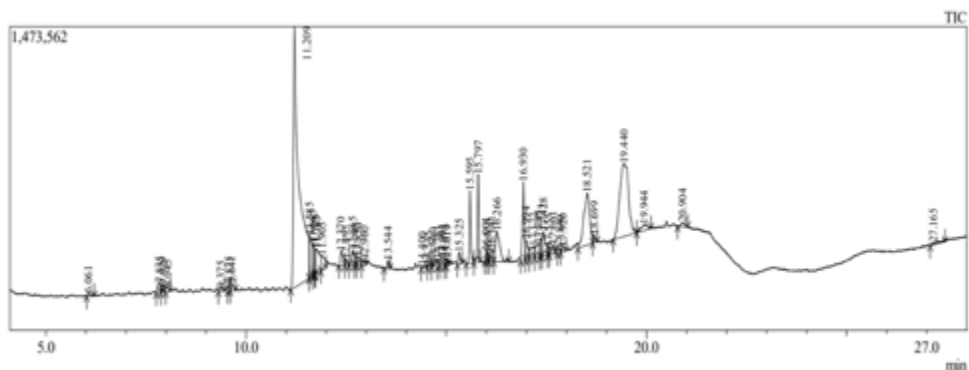


Figure 5

Total ion chromatogram of ethanol extract of Vacuum-dried RSP powder

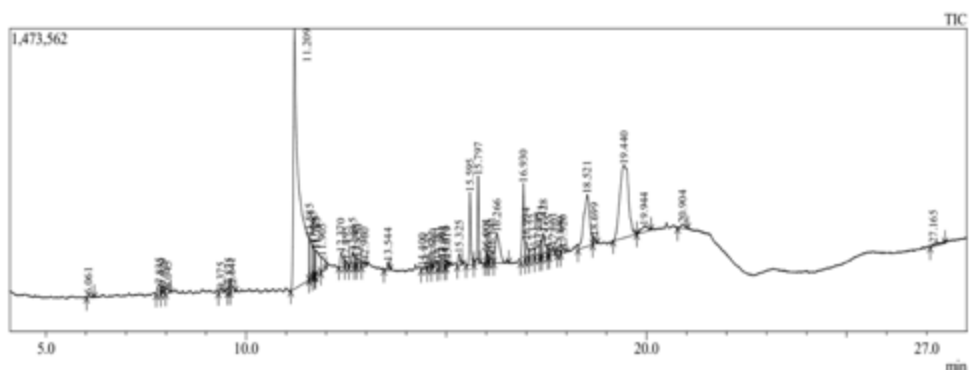


Figure 5

Total ion chromatogram of ethanol extract of Vacuum-dried RSP powder

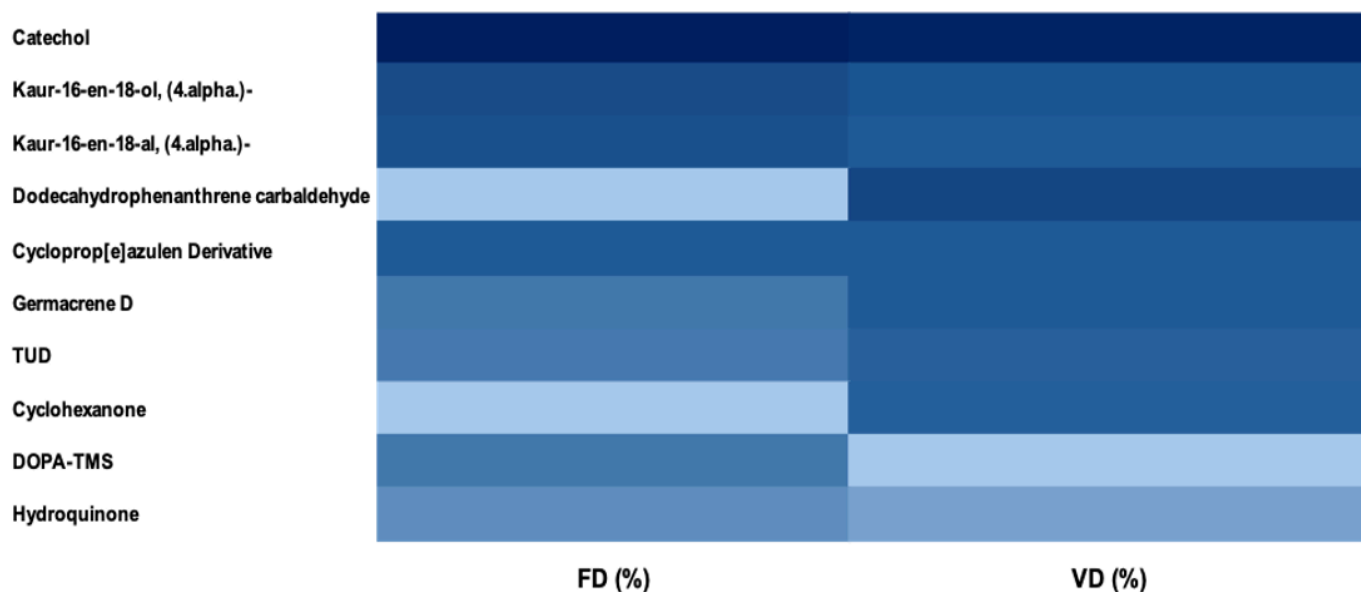


Figure 6

Heatmap comparison between the ethanol extracts of Freeze-dried and Vacuum-dried RSPs showing the normalized major GC-MS peaks after data clean-up. The values are normalized peak area % of retained compounds (rows in the left) and colour intensity corresponds to relative abundance in the FD and VD extracts.

N.B: FD: Freeze dried extract; VD: Vacuum dried extract; Dodecahydrophenanthrene carbaldehyde: (1R,4aR,4bS,7R,10aR)-1,4a,7-Trimethyl-7-vinyl-1,2,3,4,4a,4b,5,6,7,9,10,10a-dodecahydrophenanthrene-1-carbaldehyde; Cycloprop[e]azulen Derivative: 1H-Cycloprop[e]azulen-7-ol derivative; TUD: (1S,2E,6E,10R)-3,7,11,11-Tetramethylbicyclo[8.1.0]undeca-2,6-diene; Cyclohexanone: Cyclohexanone, 2-(hydroxymethyl)-; DOPA-TMS: 1-(3,4-Dihydroxyphenyl)-2-(methylanino)ethan-1-one, 2TMS

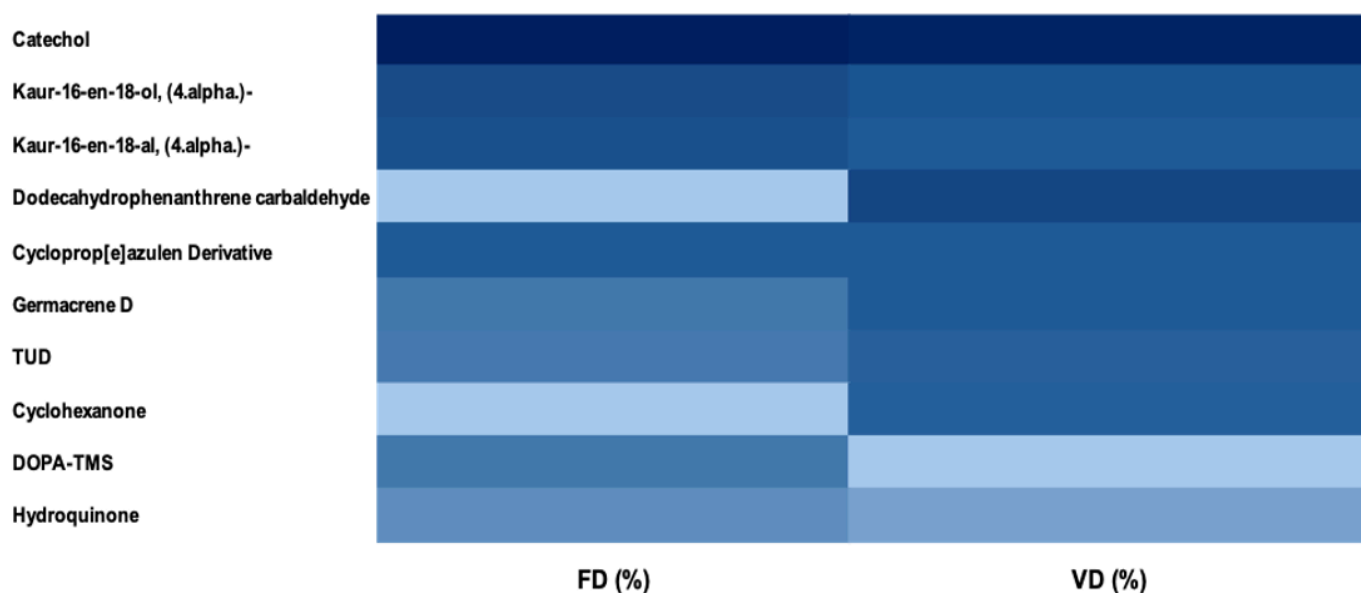


Figure 6

Heatmap comparison between the ethanol extracts of Freeze-dried and Vacuum-dried RSPs showing the normalized major GC-MS peaks after data clean-up. The values are normalized peak area % of retained compounds (rows in the left) and colour intensity corresponds to relative abundance in the FD and VD extracts.

N.B: FD: Freeze dried extract; VD: Vacuum dried extract; Dodecahydrophenanthrene carbaldehyde: (1R,4aR,4bS,7R,10aR)-1,4a,7-Trimethyl-7-vinyl-1,2,3,4,4a,4b,5,6,7,9,10,10a-dodecahydrophenanthrene-1-carbaldehyde; Cycloprop[e]azulen Derivative: 1H-Cycloprop[e]azulen-7-ol derivative; TUD: (1S,2E,6E,10R)-3,7,11,11-Tetramethylbicyclo[8.1.0]undeca-2,6-diene; Cyclohexanone: Cyclohexanone, 2-(hydroxymethyl)-; DOPA-TMS: 1-(3,4-Dihydroxyphenyl)-2-(methylamino)ethan-1-one, 2TMS

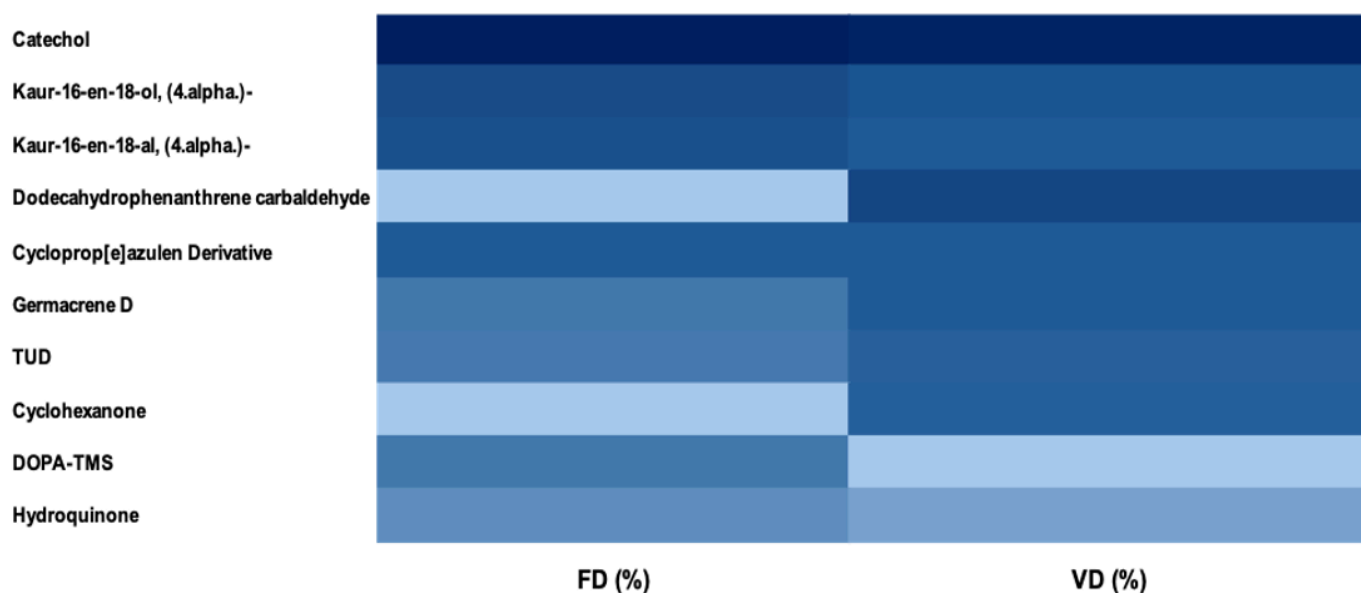


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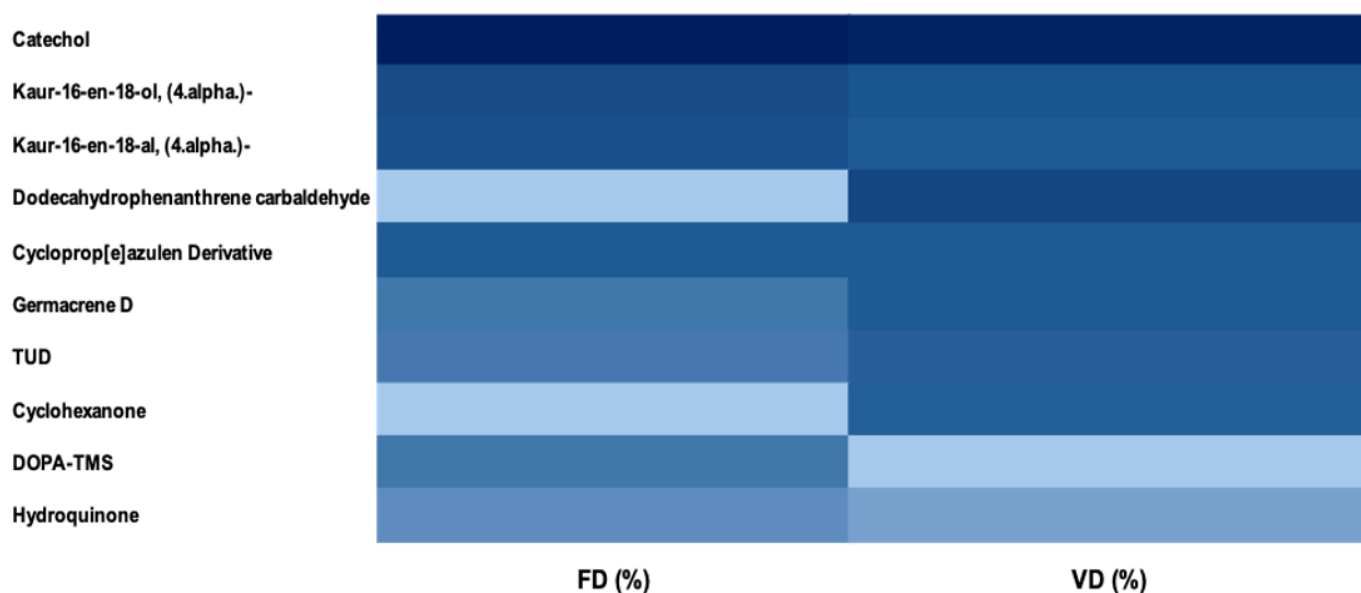


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