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Improvement of sensory quality and shelf life of fruits by applying mid-infrared

Umakanthan and Madhu Mathi

Abstract
Fruit cultivation is seasonal and geography-specific, hence preservation is needed. The use of chemical preservatives to enhance the shelf life of fruits is neither economical nor safe to health. Also safe enhancement of sensory attributes still remains a challenge. In this research we aimed to simultaneously improve the shelf life and sensory attributes of fruits using non-ionizing mid-infrared laser. We invented a device to generate 2–6 µm mid-infrared which by spraying at constant pressure ejects an imbalanced ionic solution. Jamun, strawberry, banana, mango papaya fruits were subjected to 2-6 µm mid-IR treatment. The treated fruits were found to have enhanced taste, aroma and 80-550% shelf-life. This safe, highly economical, eco-friendly, and less time consuming technology has a broad research scope and useful applicability in food industries.

Keywords: 2-6 µm Mid-IR, fruit, shelf life, sensory attributes, enhancement, economical

1. Introduction
To enhance the commercial value, fruits are treated with preservative chemicals which degrade the fruits' natural taste and aroma and lead to potential health issues. Fruit preservation without diminishing its safety and quality has been a prime challenge for food industries. The use of thermal operations such as pasteurization, drying and evaporation are still the most used methods (Rosenthal *et al*., 2018) [1]. However, energy usage made the cost of those processes increase rapidly over the last decade. De Corato U, 2019 [39], has reviewed varieties of physical, chemical and packaging methods and also combination of these methods to improve the shelf life of fruits. However, only a fewer of the solutions have gained acceptance by the food industry owing to the sensory quality being affected. Thus, new technologies are being researched with the goal of finding new preservation methods using less energy but still providing the safety and quality required.
Infrared is gaining popularity in the recent years because of the thermal efficiency when compared to traditional technologies. Application of infrared includes pasteurization of several food products, but also has potential for blanching, dehydration, roasting, baking and cooking (Rastogi, 2012) [2]. Infrared transfers energy in the form of electromagnetic waves and thus less time for heat transfer is required (Yu *et al*., 2017) [3]. The majority of applications of IR are found in mid-infrared region of the spectrum region. According to Lu *et al*., 2011 [4], the mid-IR spectra can be divided into four major regions. Aliphatic C—H stretching modes absorb in 3000–2800 cm^{-1} (region 1); although most components of food (proteins, carbohydrates, etc.) contain aliphatic C—H groups, this region is most frequently correlated with fatty acids. Region 2 (1800–1500 cm^{-1}) contains the C═O stretching band of lipids at ca 1740 cm^{-1} and the amide I and II bands of proteins and peptides at ca 1650 and 1550 cm^{-1}, respectively. The amide bands provide structural information about α-helix, β-sheet and random coil conformations in proteins. Many cellular components have absorption bands between 1500 and 1200 cm^{-1} (region 3), for example, nucleic acids and phospholipids. The spectral interval between 1200 and 900 cm^{-1} (region 4) provides information about the structure of polysaccharides.
Most fruit components contain molecular groups that absorb energy from the mid-IR region. Some works have studied the effects of IR heating on the shelf life of foodstuffs (Doymaz, 2018) [5]. However, there is still lack of knowledge on the effects of mid-IR on powdered fruit and the effects of IR on microbial inactivation and product quality (Staack *et al*., 2008) [6] (which probably lost/ diminishing due to our modern agricultural practice).

Thus, the objective of our work was to restore/ enhance the natural taste and aroma of fruits, and enhance their shelf-life using our newly invented mid-infrared generating atomizer (MIRGA) technology.

2. Materials and Methods
2.1 Materials
Powdered Banana (*Musa acuminate*), Strawberry (*Fragaria ananassa*), Jamun (*Syzygium cumini*), Mango (*Mangifera indica* L.) and Papaya (*Carica Papaya* L.) were bought at a local market.

2.2 Mirga apparatus
Specifications of mid-infrared generating atomizer (MIRGA) *(patent no.: 401387)* (Fig 1) and the process of generating mid-IR while spraying MIRGA are described by Umakanthan *et al*., 2022a, Umakanthan *et al*., 2022b [7, 8]. Spraying should be carried out at a distance of 0.25 - 0.5 m toward any type of packaged (polythene) fruits. Every spraying depending on plunger pressure generates 2-6μm mid-infrared. The inorganic compounds used in the generation of MIR are a perspective for biomedical applications (Tishkevich *et al*., 2019; Dukenbayev *et al*., 2019) [9, 10]. It is also a new synthesis method for preparation of functional material (2-6 μm mid-IR) (Kozlovskiy *et al*., 2021; El-Shater *et al*., 2022) [11, 12]. It is well known that the combination of different compounds, which have excellent electronic properties, leads to new composite materials, which have earned great technological interest in recent years (Kozlovskiy *et al*., 2021; Almessiere *et al*., 2022) [13, 14].

Ethical statement and Informed consent: Since spraying was only external, institutional ethical review board deemed sensory ethical approval as unnecessary. Informed consent was obtained from all the participants.

2.3 Mirga Trial I with fresh fruits
Sixty 200g packets (polyethylene with 70μm thickness) of Jamun fruit was prepared and sealed with cellophane. Twenty packets were one time MIRGA sprayed and marked 1. Another 20 packets were sprayed twice and marked 2. The remaining 20 packets were controls (non-sprayed) and marked C. Ten packets from each 1, 2 and C (n=30 total) were kept at room temperature and the remaining 30 in a domestic refrigerator. The 1, 2 and C fruits were subjected to a sensory expert panel test (n=6) immediately after treated and during the preservation in both room temperature and refrigeration. Subsequently, fruits (1, 2, and C) were periodically subjected to sensory panel tests. The acceptability index used was a hedonic scale with 9-point nominal structure: 1 - Dislike extremely, 2 - Dislike very much, 3 - Dislike moderately, 4 - Dislike slightly, 5 - Neither like nor dislike, 6 - Like slightly, 7 - Like moderately, 8 - Like very much, 9 - Like extremely (Everitt, 2009; Wichchukit *et al*., 2014) [15, 16].

The same procedure was adopted individually for all the trailed fresh fruits. Trials were repeated 6 times.

2.4 Mirga Trial II with fruit powders
Polythene-packed (thickness above 51μm) marketed Jamun, banana, and mango powders were purchased. Immediately, a control sample was taken and subjected to sensory expert panel tests (the control).Then, after every spraying fruit powders were subjected to sensory evaluation and spraying was continued until the powders become more palatable (but later become unpalatable on more spraying). The control samples, the jamum samples after 5 and 13 sprays, the banana samples after 3 and 10 sprays and the mango samples after 3 and 9 sprays were subjected to Gas chromatography–mass spectrometry (GC-MS), Fourier-transform infrared spectroscopy (FTIR), Powder X-ray diffraction (PXRD), Proton nuclear magnetic resonance (1H-NMR), and Transmission electron microscopy (TEM). These samples were chosen because they presented the extremes between palatable and unpalatable. Trials were repeated 6 times. (i.e., jamun 5 and 13, banana 3 and 10, and mango 3 and 9 sprayings)

3. Results and Discussion
3.1 Mid-infrared emissions
Five fruits were sprayed with mid-infrared generated from the atomization setup. Fig F2 presents the estimated 2-6 μm mid-infrared emitted from MIRGA. This distance is essential for the MIRGA-sprayed solution to form ion clouds, oscillations, and mid-IR generation. The ray can penetrate the intervening packaged material and act on the fruits inside. Close spraying doesn't generate energy. Thus, MIRGA should be operated/ used like a body spray, i.e. externally over any packed/unpacked material.

3.2 Shelf life of sprayed fruits
The shelf-life of sprayed fruits was determined by sensory panels. Fresh fruits were sprayed for 1 and 2 times to enhance the taste, aroma and palatability. Then the sensory panel tested the fruits within 2-5 minutes after each MIRGA spraying. The results are presented in Supplementary Table 1.

Analysing the results, the shelf life of sprayed fruits was extended by 80-540%. The fruit most susceptible to shelf-life increasing is the Strawberry while the Papaya is the least improved by the method. Moreover an increase in the shelf life was observed when fruits are stored in a refrigerator, when compared to room temperature storage.

When comparing the results between 1 and 2 sprays, one time spraying presented an increase in shelf life. The reason for more spraying rounds in fruit powders is that extra energy in nature should denature the target's inherent characters. We did this until the fruit powders' inherent characters were almost or completely lost.

The trials were repeated in the fruits, marketed and grown at different geographical area, and the results were found to be almost same with negligible difference.

3.3 Multiple spray treatment and Sensory scoring
Results revealed that jamun, banana and mango powders had enhancement of taste, aroma and palatability after 5, 3 and 3 sprayings, respectively. Alternatively, unpalatability occurred after 13, 10 and 9 sprayings respectively (Table 2). These sensory attribute changes were perceived in 2-5 minutes after spraying.

3.4 Instrumentation Results: (Data file D1)(instrumentation details in Section S(I))

3.4.1 GC MS
a) GC MS – Banana: (Detailed interpretation in Supplementary file Section S(II))

Control contains many ester forms of fatty acid such as Hexadecanoic acid, and other molecules such as *tert*-Hexadecanethiol, Hexadecane, 1,1-bis(dodecyloxy), etc. The major fatty acid was Hexadecanoic acid. In 3 sprayed sample there was new peak of Methane, oxybis[dichloro] and D-Allose. In addition, there was decrease in peak of Hexadecanoic acid. Also, there was peak of trans fatty acid which have food and clinical significance. This is responsible for enhancement of sweetness and aroma characters. 10 sprayed sample has shown unique peak of 9-Octadecenamide and Nonadecane, 9-methyl which is responsible for tastelessness with sweetness reduction and hence non-palatable. D-Allose was the most abundant peak seen in 10 sprayed sample with decrease in peak of Hexadecanoic acid as compared to control. One more observation was seen that after 3 and 10 sprayings, the fatty acid ester was changed into its corresponding fatty acids. (Fig 2a) **(**Datta *et al*., 2014; Xu *et al*., 2017) [17, 18]

b) GC MS – Mango (detailed interpretation in Supplementary file Section S(III))

Control sample contains many fatty acids, such as Hexadecanoic acid, and other molecules such as 2,5-Furandione, 3-methyl, 2-Thiazolamine, 4,5-dihydro, etc. There are unique peaks formoleic acid& n-Hexadecanoic acid, which are disappearing after MIRGA sprayings. In 3 sprayed sample, there was not peaks of fatty acids at all and possessed all other peaks present in control. Further several peaks such as 2,5-Furandione, 3-methyl, 2,4-Dihydroxy-2,5-dimethyl-3(2H)-furan-3-one, 2-Thiazolamine, 4,5-dihydro were increased as compared to control sample. These changes in 3 sprayed sample is responsible for faster dissolving in the mouth (Esmaeili, 2015) [23]. 9 sprayed sample has shown a unique peak of 1, 8:2, 7-Dimethanodibenzo [a,e]cyclobuta[c]cycloocten-13-one and Tetradecanoic acid. Also, there was considerable increase in peak of 2-Furancarboxaldehyde, 5-(hydroxymethyl) which is responsible for reduction in sourness, aroma, and tastelessness. 9 spraying has resulted in the reduction of fatty acid (n-Hexadecanoic acid and oleic acid) peak, which is the reason for a long time for dissolution in the mouth (Atkins *et al*., 2011) [19]. (Fig 2b)

3.4.2 FTIR

a) FTIR – Jamun: (Detailed interpretation in Supplementary file Section S(IV))

In general, compared to control, peaks area reduced in 5 sprayed and 13 sprayed samples. In 5 sprayed sample partial destruction of long-chain structures took place, while carbohydrate structures did not suffer much destruction. In 13 sprayed sample, destruction of protein structures with the formation of amino acids occurred, and hydrocarbon structures also underwent partial destruction (Datta *et al*., 2014) [17]. (Fig 3a)

b) FTIR – Banana: (Detailed interpretation in Supplementary file Section S(V))

The peak around 3386 cm^{-1} in control has shifted to around 3415 cm^{-1}in the 3 sprayed spectrum. In addition, the peak width is reduced. It is suggested that an increase in the number of compounds with N-H bonds are reduced hydrogen bonding (due to the reduced peak width) (Mohan, 2004) [20] which are responsible for increased sweetness, taste, and aroma of the 3 sprayed sample compared to the control. In addition, the peak at 854 cm^{-1} (C=C bending) is reduced in the 3 sprayed sample compared to control. This reduced amount of compounds containing C=C bonds are responsible for the improved flavor.

10 sprayed sample spectrum shows a peak at 436 cm^{-1}; whereas, the control and 3 sprayed samples spectra do not indicate this peak. The peak indicates there are complex deformations of many compounds and this lead to the tastelessness, reduced sweetness and reduced aroma. (Fig 3b)

c) FT IR – Mango (Detailed interpretation in Supplementary file Section S(VI))

Though the spectra show peaks at similar wavenumbers (cm^{-1}) and with similar shape and intensity, but N-H, O-H, C=O, C=C stretching **(**Alvarez *et al*., 2012; Shankar, 2017) [21, 22] are prominent in the sprayed sample corresponding to the spraying number. (Fig 3c)

3.4.3 PXRD

a) PXRD – Jamun: (Detailed interpretation in Supplementary file Section S(VII))

5 sprayed shows the highest relative intensity of all the peaks identified. Split peaks are present in all the samples between 17° and 18°. These split peaks are most prominent and easily separated in 5 sprayed, followed by 13 sprayed. 5 sprayed has the highest volume-percent of crystalline phases as shown by the number of peaks present. 5 sprayed has additional peak in 36.6° while 13 sprayed has additional peaks at around 4.97°, 8.82° and 60.18° indicating the formation of new crystalline phases aside from those already existing in control (Esmaeili, 2015) [23]. (Fig 4a)

b) PXRD – Banana: (Detailed interpretation in Supplementary file Section S(VIII))

The three spectra exhibited prominent peaks at comparatively same directions. Peak intensities of 3 and 10 sprayed samples, are relatively lower than that of the control. 3 sprayed has split minor peaks at low angle 2θ which are absent in control and 10 sprayed. This scenario indicates formation of new phase in 3 sprayed sample. (Fig 4b)

c) PXRD – Mango: (Detailed interpretation in Supplementary file Section S(IX))

Peak of control around 15.07° splits in 3 sprayed and 9 sprayed. New peaks were observed around 26° and 31° for both 3 sprayed and 9 sprayed. Some of these new peaks are noticeably narrower and more intense relatively. Minor peaks observed around 5° of control and 9 sprayed is absent in 3 sprayed. These scenarios indicate formation of new phases in sprayed samples. (Fig 4c)

3.4.4 TEM

a) TEM – Jamun

Control: Amorphous/microcrystalline texture. Nanoparticles 5-10 nm semi spherical. Particles 100-200nm ellipsoidal or amorphous. Fibres 1-2 µm length, thin filament. Aggregates >1nm amorphous. Smooth structure. 5 sprayed sample: Amorphous/microcrystalline texture. Only aggregates seen, size is 1µm and amorphous in shape. Smooth structure. 13 sprayed sample: Amorphous/microcrystalline texture. Again nano particles appeared 10-20 nm semi spherical. Particles 100-200nm ellipsoidal or amorphous. Fibres are very variable in length, thin filament, evenly spread and strongly intersected each other. Aggregates >1nm amorphous. Nano particles and fibres appeared in large number. Structure not smooth but sponge like. (Fig 5a)

Increasing the spraying number causes significant alteration of the sample texture; peculiarly, this concerns more in 5 sprayed than 13 sprayed samples. (Datta *et al*., 2014) [17].

b) TEM – Banana

Diffraction patterns: Control banana powder shows amorphous (or microcrystalline) atomic arrangement. 3 spraying caused atomic arrangement leading to crystalline structure. Whereas 10 spraying again caused atomic arrangement leading to amorphous (or microcrystal) form.

Bright field images: With respect to the control, 3 and 10 spraying induced significant differences in the matrix structure; moreover, the effects greatly differ with spraying itself. In particular, 3 spraying caused a crystalline atomic arrangement of the sample, whereas the control is amorphous, and the 10 spraying did not caused significant changes in the atomic arrangement as well. Other relevant differences concern both aggregates and nanoparticles components. In the first case, the circular structure of aggregates in the control is completely lost in both 3 and 10 sprayed samples, which in turn show aggregate types differing between the two samples. In the second case, nanoparticle numerosity decrease progressively from the control to the 10 sprayed sample; moreover, significant changes are observed from control (nanoparticles show uniform distributions of shape, size, orientation and spatial arrangement, and are non-crystalline), to 3 sprayed sample (nanoparticles show uneven distributions of the above cited features, reduced number, and are crystalline), to 10 sprayed sample (nanoparticles presence is negligible). (Fig 5b).

c) TEM – Mango

Electron diffraction pattern: With respect to the control, 3 and 9 sprayings induced significant differences in the matrix structure and a loss of the polycrystalline arrangement of the control, since both 3 and 9 sprayed samples are amorphous.

Bright field images: In addition, 3 spraying affected the presence of aggregates, and the nanoparticle structure (loss of squared shape observed in control, different spatial arrangement, larger size variability, uneven mass distribution within particle), while 9 spraying affected the nanoparticles component (only the 10 – 30 nm size fraction is observed, differently from control and 3 sprayed samples) and its spatial arrangement (nanoparticles are only observed in large clusters, differently from control and 3 sprayed samples). (Fig 5c)

Similar desirable results in coffee, tea, cocoa and edible salts were achieved using MIRGA spraying by Umakanthan *et al*., 2022a; Umakanthan *et al*., 2022b; Umakanthan *et al*., 2022c [7, 8, 24].

4. Mode of action of mid-IR on fruits: (Detailed discussion in Supplementary file Section S(X))

Umakanthan *et al*., 2022a [7], has discussed about the MIRGA's invention background, definition, technique of mid-IR generation and its safety studies using *in-vitro* Vero, A549 and Human dermal fibroblast cells. The action of MIRGA emitted 2-6 µm mid IR on the fruits are discussed below.

While spraying MIRGA, the 2-6 µm MIR generated from the MIRGA scatters through the air and gets absorbed by receptors (fruits) molecules and caused chemical and molecular level changes in the fruits. The mid-infrared caused photodegradation, i.e. alterations of materials by light; thereby the fruit molecules are degraded or transformed into another molecule/ compound, as reported in GCMS analysis. Yousif *et al*., 2013 [40], has explained this process as photodissociation of molecules caused by the absorption of photons of sunlight, such as infrared radiation, visible light, and ultraviolet light leading to change of a molecule's shape. Moreover, nowadays generally flavours of fruits are decreasing. Flavor involves a combination of sugars, acid and volatile acids. Consumers concern more on flavor. Scientifically it is difficult to enhance the falvor. And Transgenic and molecular assisted breeding to improve the flavor is also difficult. Also flavor quality is a complex trait and chemical addition to improve flavor is risky and usually consumers dislike.

Earlier shelf life of fruits has been improved using irradiation with ultraviolet (Antonio *et al*., 1998) [26], infrared (Vishwakarma *et al*., 2013) [27] and gamma (Ehab *et al*., 2014; Antaryami *et al*., 2016; Amee *et al*., 2018; Ehab *et al*., 2018) [28, 30, 29, 31] radiation. Other technologies improve shelf life involved the use of 2,4-epibrassinolide ethanol solution (Zhang *et al*., 2017) [32], films with chemical additives and polymer (Sareen *et al*., 2019) [33], nano chitosan-based coatings (Kondle *et al*., 2022) [34] and evaporative cooling system (Ayobami *et al*., 2023) [35].

Fewer studies were done on the improvement of sensory attributes in fruits *viz*., heat and calcium treatment (Joshua *et al*., 1998) [36], food transformation, enzymatic and acidic hydrolysis, postharvest heat treatment, and use of edible coating film (Louisa *et al*., 2020) [37], and addition of active edible coating (Aswini *et al*., 2022) [38]. Fruits shelf life and sensory attribute improvement are costlier. Literature review showed that except MIRGA no technology is found available for concurrent improvement of shelf life and sensory attributes.

Table 1: Effects of MIRGA on shelf life of five different fresh fruits

Samples used	Number of MIRGA sprays	Number of days without spoilage at room temperature		Shelf life increase %	Number of days without spoilage in a refrigerator		Shelf life increase %
		Control	Trial		Control	Trial	
Jamun	1	2	8	300	5	9	80
Strawberry	1	2	20	900	5	32	540
Banana	1	4	12	200	6	27	350
Mango	1	7	20	185	8	37	362
Papaya	1	10	19	90	10	33	230
Jamun	2	2	9	350	5	13	160
Strawberry	2	2	13	550	5	28	460
Banana	2	4	14	250	6	26	333

Mango	2	7	15	114	8	35	337
Papaya	2	7	16	128	10	30	200

Table 2: Sensory profiling of Fruits

No. of sprays	Fresh fruits					Fruit powder		
	Jamun	Strawberry	Banana	Mango	Papaya	Jamun	Banana	Mango
Control	2	2	2	2	2	2	2	2
1	6	6	7	6	7	3	4	3
2	7	6	6	7	6	5	6	5
3	-	-	-	-	-	5	6	7
4	-	-	-	-	-	6	7	6
5	-	-	-	-	-	7	7	5
6	-	-	-	-	-	6	5	3
7	-	-	-	-	-	6	4	3
8	-	-	-	-	-	5	3	2
9	-	-	-	-	-	5	2	1
10	-	-	-	-	-	4	1	-
11	-	-	-	-	-	3	-	-
12	-	-	-	-	-	2	-	-
13	-	-	-	-	-	1	-	-

Hedonic score description: 1 - Dislike extremely, 2 - Dislike very much, 3 - Dislike moderately, 4 - Dislike slightly, 5 - Neither like nor dislike, 6 - Like slightly, 7 - Like moderately, 8 - Like very much, 9 - Like extremely.

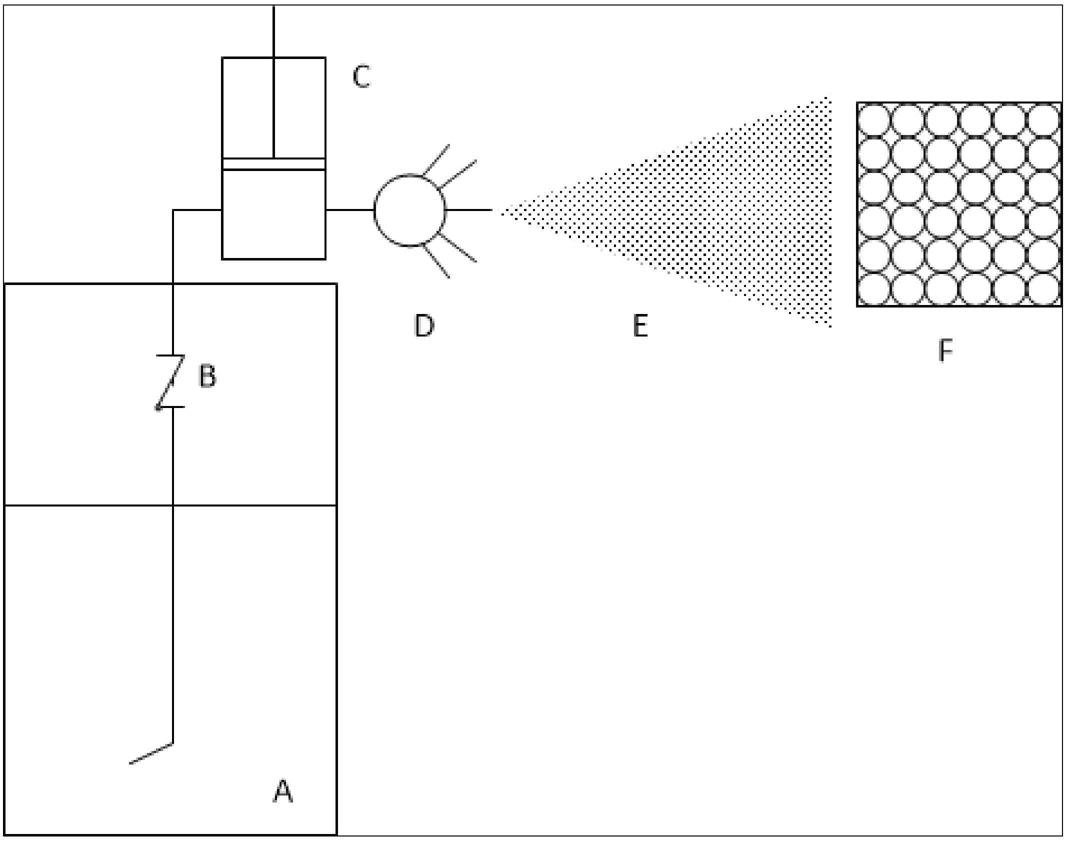


Fig 1: Schematic apparatus of MIRGA (A) Spraying solution tank; (B) Valve; (C) Pressure Pump; (D) Nozzle; (E) Mid-IR Spray; (F) packed and sealed fruits

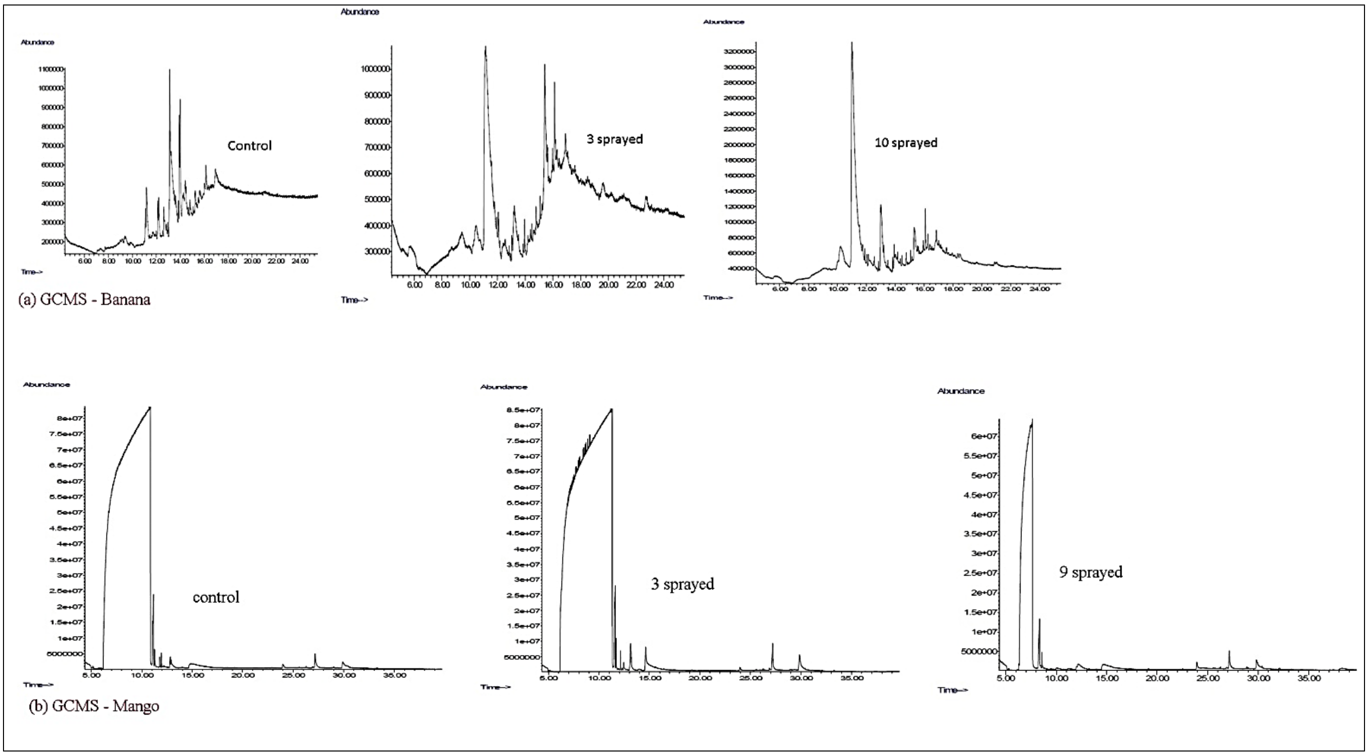
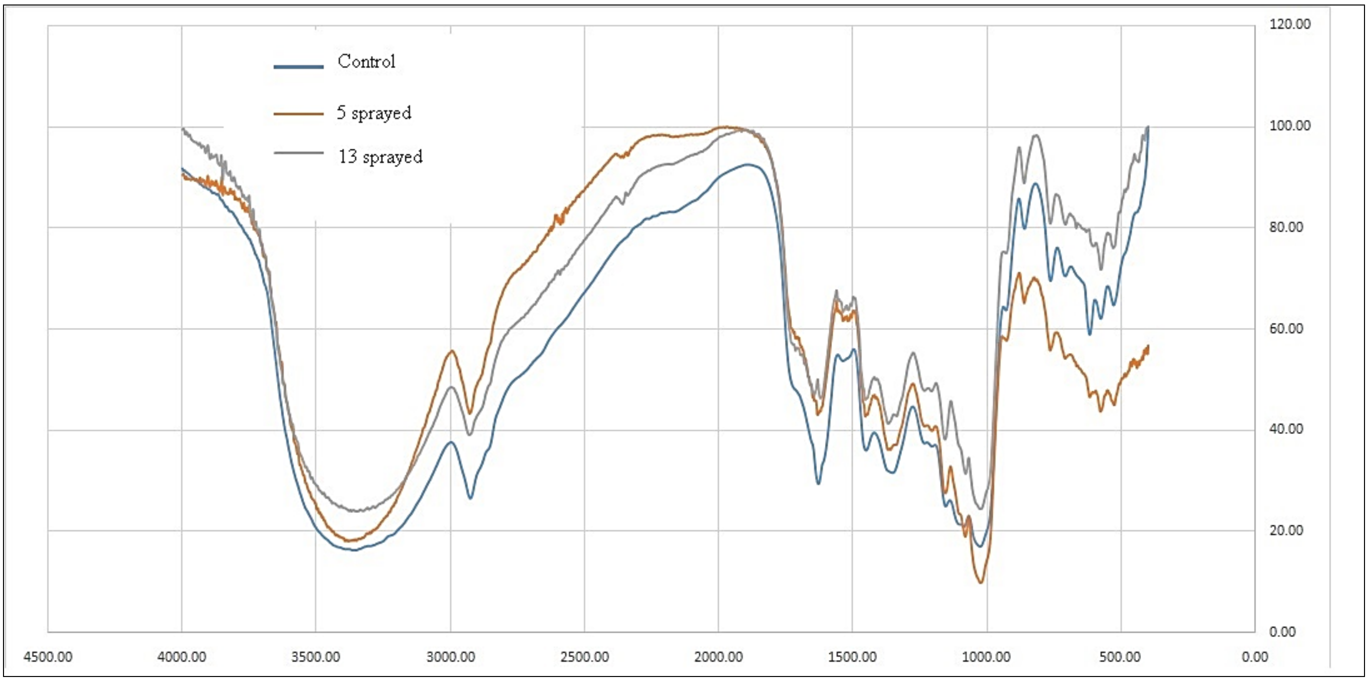
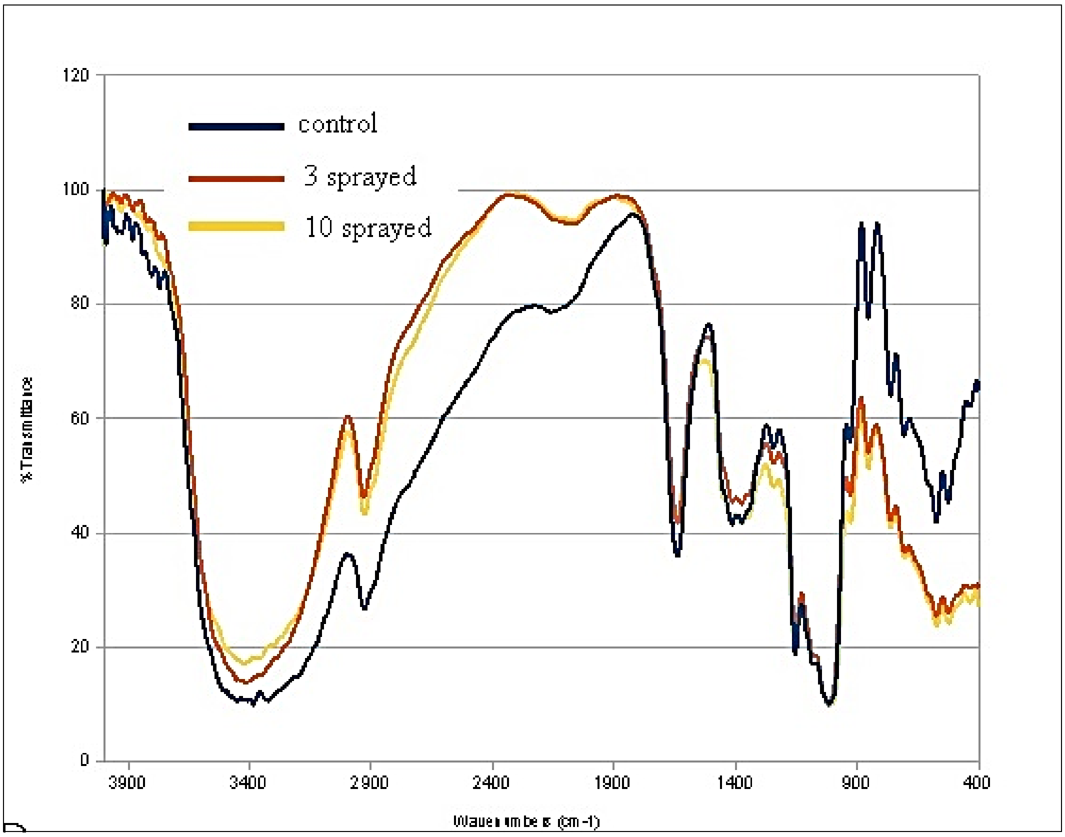


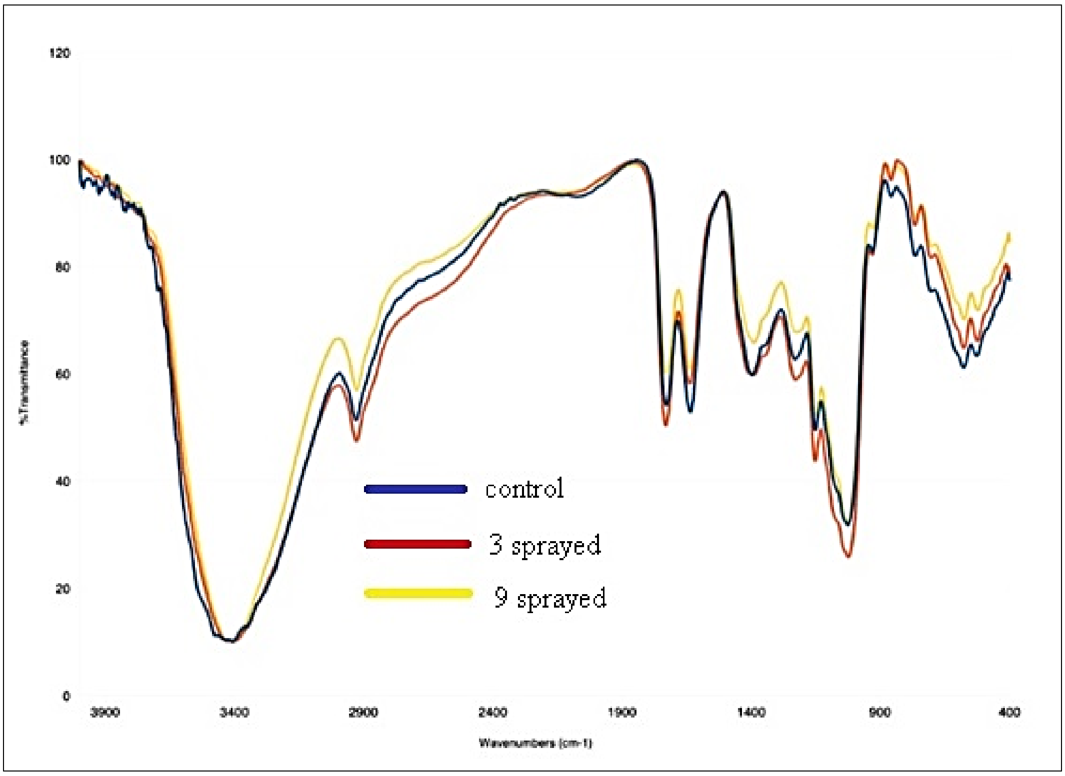
Fig 2: GCMS spectra (a) Banana, (b) Mango



(a) FTIR - Jamun

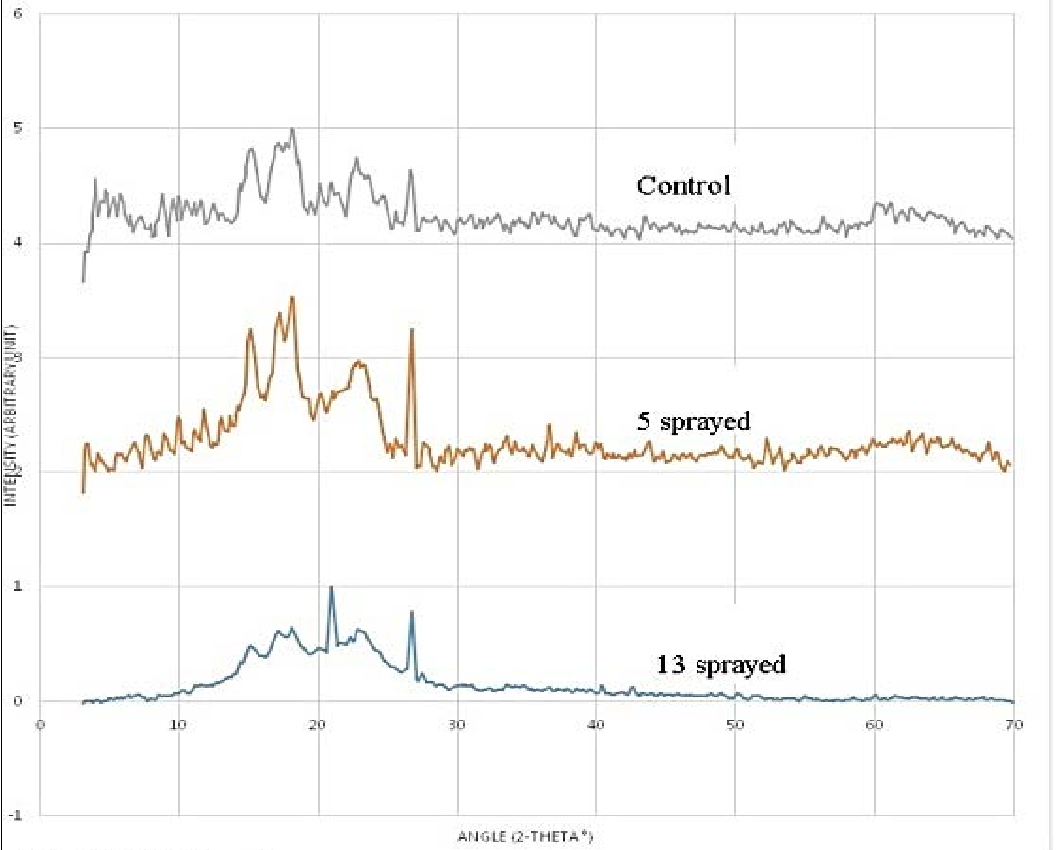


(b) FTIR - Banana

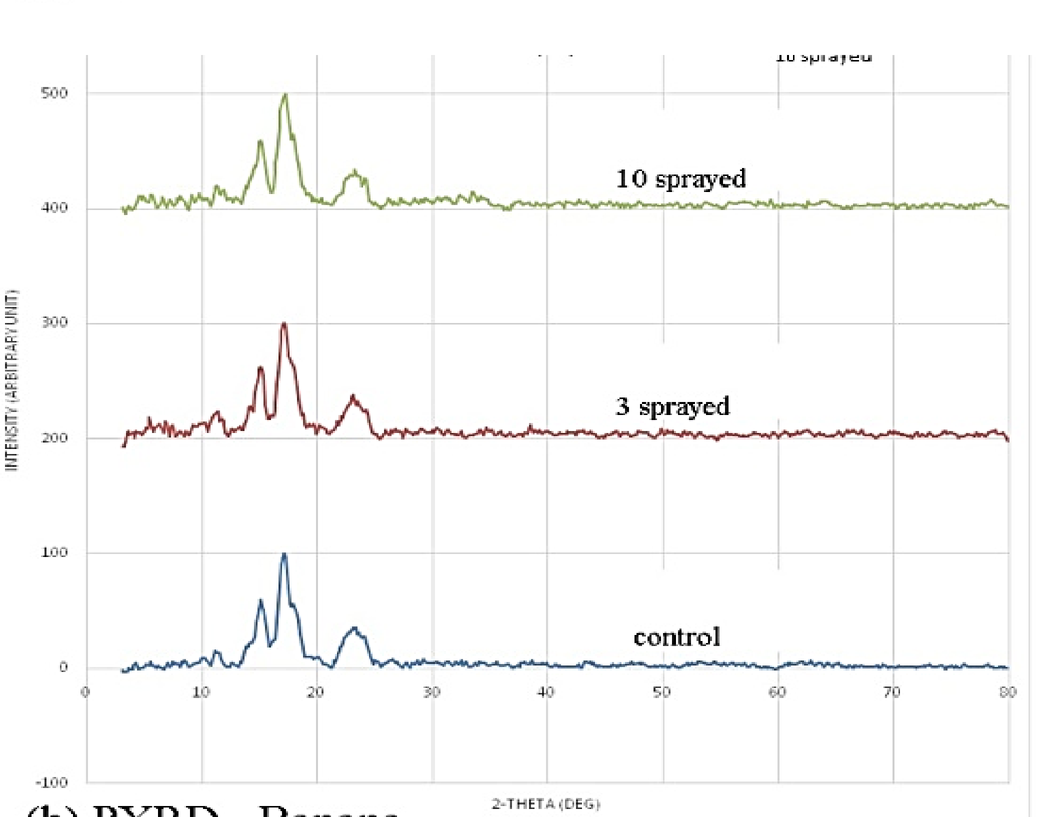


(c) FTIR - Mango

Fig 3: FTIR spectra (a) Jamun, (b) Banana, (c) Mango



(a) PXRD - Jamun



(b) PXRD - Banana

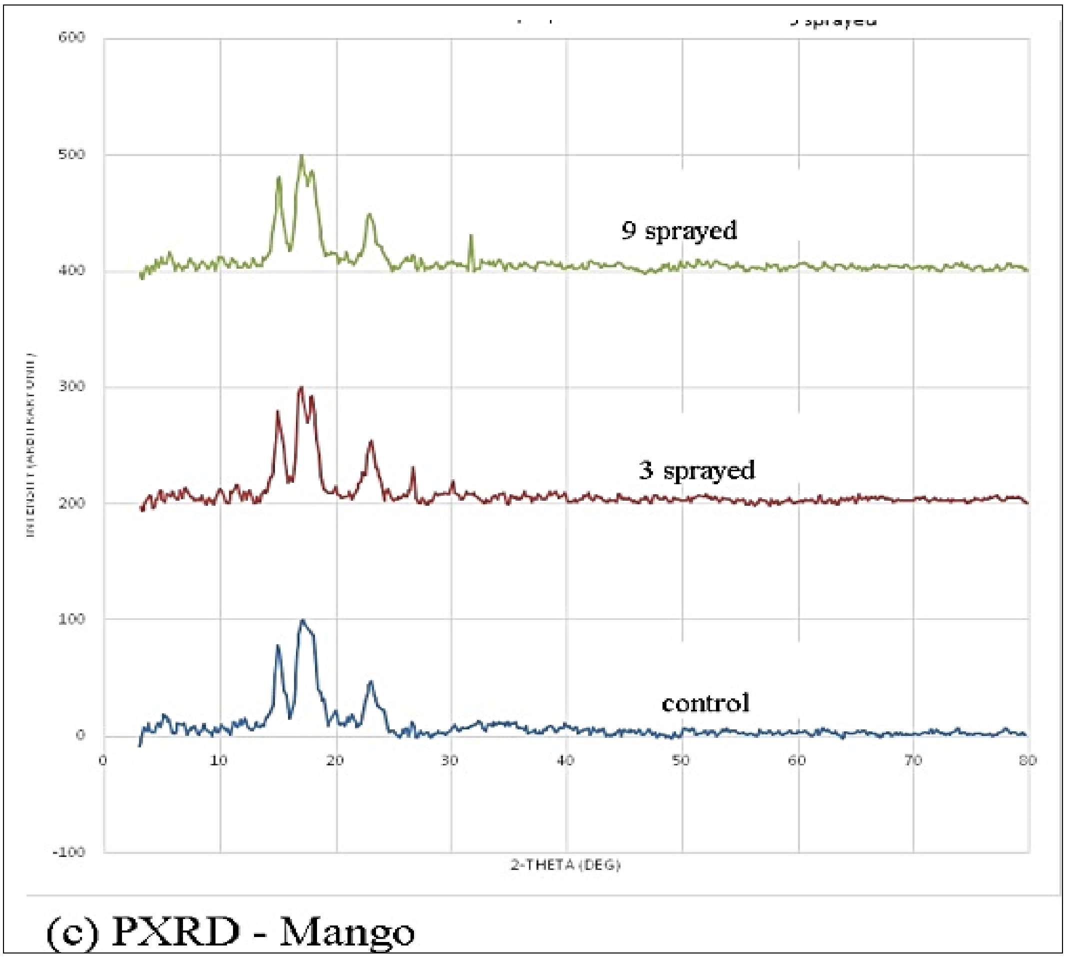
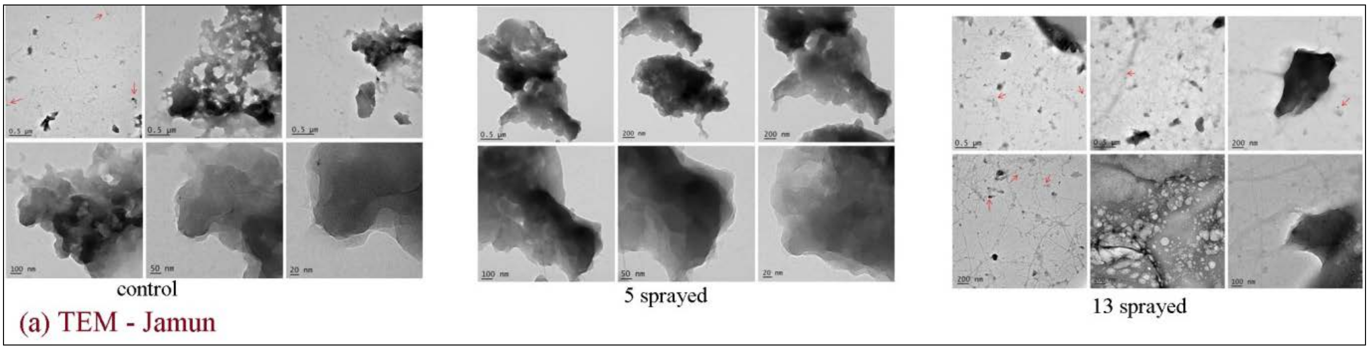
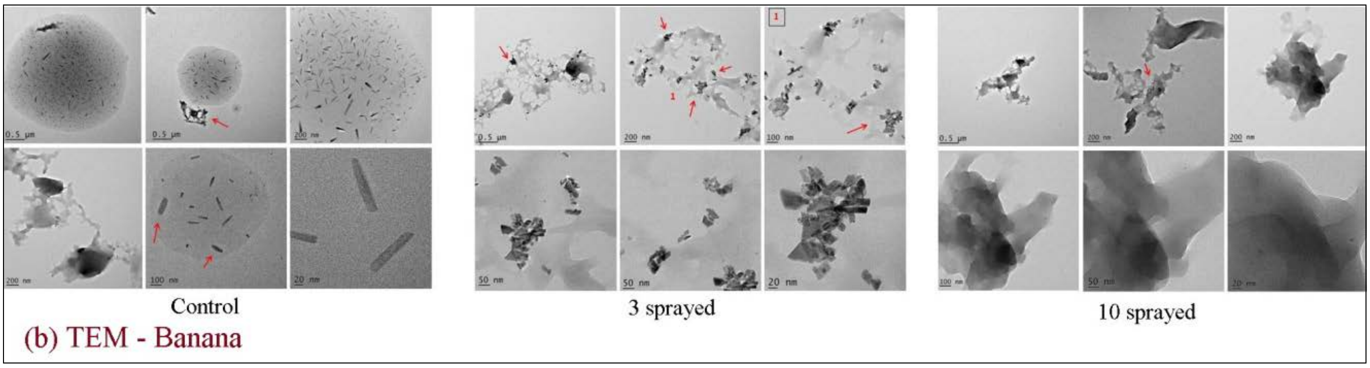


Fig 4: PXRD spectra (a) Jamun, (b) Banana, (c) Mango





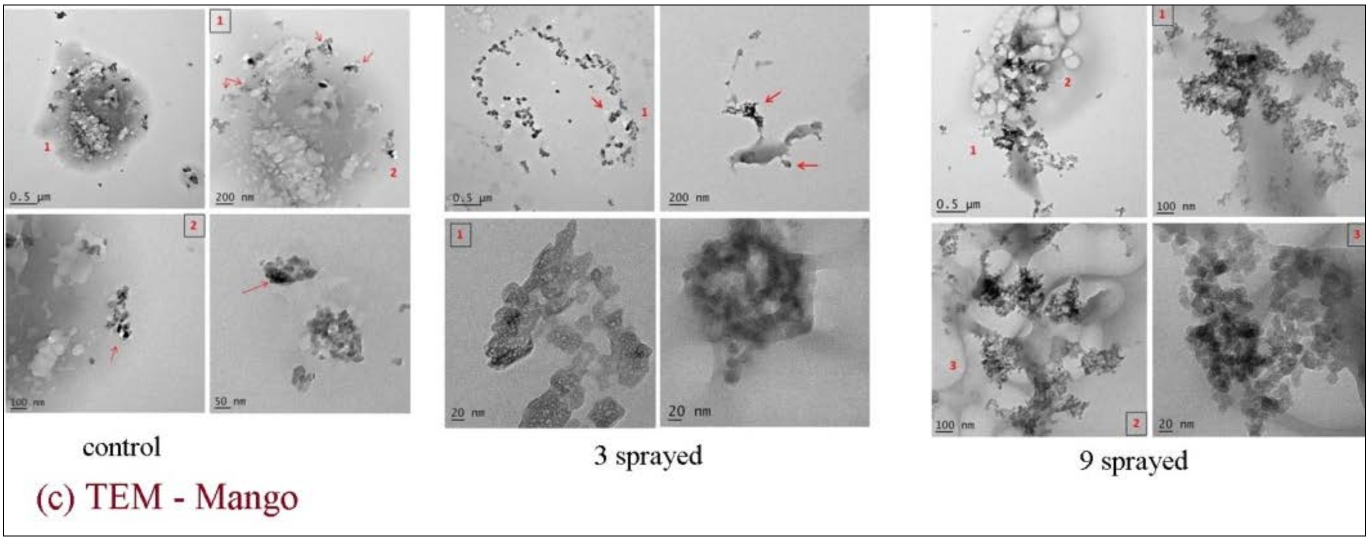


Fig 5: TEM – bright field images (a) Jamun, (b) Banana, (c) Mango

5. Conclusion

Five different fruits were successfully subjected to the safe mid-IR which increased its aroma, flavor, palatability and shelf life. The mid-IR technology developed in the present work can be beneficial for other fruits sensory traits enhancement and preservation. Finally, the inclusion of mid-IR can be beneficial for industries because it is a low cost technology with great economic benefit, and easy to operate.

6. Supplementary file available in

https://docs.google.com/document/d/1ikPOTBQAMu7VFUxx0kvnWEuXlwmrKdPE/edit?usp=sharing&ouid=111101387151809704391&rtpof=true&sd=true

7. Author contributions

Umakanthan: Conceptualization, Methodology, Resources, Supervision, Funding.
Madhu Mathi: Investigation, Data curation, Visualization, Writing - Original draft preparation.
Umakanthan and Madhu Mathi: Project administration, Validation, Writing- Reviewing and Editing.

8. Conflict of interest

In accordance with the journal's policy and our ethical obligation as researchers, we submit that the authors Dr. Umakanthan and Dr. Madhu Mathi are the inventors and patentee of Indian patent for MIRGA (Granted-patent no.: 401387) which is a major material employed in this study.

9. Funding: This study received no specific funding.

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