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Effect of Mid-Infrared Irradiation on Laboratory Chemicals, and Scope for Future Benefits

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Abstract
Laboratory chemicals are synthesized, isolated and/or purified to suit a wide variety of purposes from industry to research labs. There are some methods for potentiation of pharmaceuticals viz., additives, synergistics but not in lab chemicals. Potentiation of lab chemical would reduce its quantity requirement, cost and saving of resources. We invented a 2-6 µm mid-infrared generating atomizer (MIRGA). Laboratory chemicals were e.g. sodium chloride, sodium bicarbonate, and anhydrous D-(+)-glucose were irradiated with MIRGA and found to be potentiated. The trialed chemical compounds were subjected to instrumentation to study the irradiation effect on their chemical bonds, structure, configuration besides sensory attributes. In the future, this economical, fast, easy and safe method may be useful in modifying the quantity of chemicals required for various purposes.

Keywords: MIRGA, 2-6µm mid-infrared ray, laboratory chemicals, irradiation, natural characteristics, potency, enhancement, economy, resource savings, safe

Introduction
An important global market, the laboratory chemicals industry is significantly growing every year, and it holds a major position in scientific technologies. Laboratory chemicals are segmented according to the purpose for which they are utilized by the end users. Further, laboratory chemicals with enhanced potency could help drive the continual advancement of technologies and their demand but no research done yet. To this end, we employed mid-infrared generating atomizer (MIRGA) to try to alter the physicochemical properties and inherent characteristics of some laboratory chemicals.

Materials and Method
MIRGA: MIRGA *(patent no.: 401387)* is a 20-mL capacity polypropylene plastic atomizer containing an inorganic (molar mass 118.44 g/mole) water-based solution in which approximately two sextillion cations and three sextillion anions are contained. The sprayer unit has dimensions 86 × 55 × 11 mm, an orifice diameter of 0.375 mm, ejection

volume 0.062 ± 0.005 mL, and ejection time 0.2 s. The average pressure is 3900 Pa, and the cone liquid back pressure is 2000 N/m^2. During spraying, approximately 1-µg weight of water is lost as mist and the non-volatile material in the sprayed liquid has a concentration of 153 mg/mL. Every time spraying emits 0.06ml which contains approximately seven quintillion cations and eleven quintillion anions. Depending on the pressure applied to the plunger, every spraying is designed to generate 2–6 µm as estimated by an FTIR (retro-reflector) interferometer instrument (Detector type D* [cm HZ1/2 - 1] MCT [2-TE cooled]) at Lightwind, Petaluma, CA, USA. Design of the MIRGA and emission of 2-6µm mid-IR has been presented in detail by Umakanthan *et al*., 2022a; Umakanthan *et al*., 2022b; Umakanthan *et al*., 2023c; Umakanthan *et al*., 2023d [1-4].

Perspectives of MIRGA: The inorganic compounds used in the generation of mid-IR are a perspective for biomedical applications [5, 6]. It is also a new synthesis method for preparation of functional material (2-6 µm mid-IR) [7, 8]. 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 [9, 10].

Sodium bicarbonate, sodium chloride and anhydrous D-(+)-glucose were purchased from an Indian laboratory. A trained 6-member sensory expert panel was employed for sensory evaluation of the laboratory chemicals.

The instruments used in this research to determine:

Chemical compound transformation – gas chromatography mass spectrometry (GC-MS): Agilent technologies, 7820 GC system, 5977E MSD, Colomn DB-5, Over temp 100-270^{0}C, Detector MS, Flow rate 1.2, Carrier gas Helium.

Chemical bond changes–fourier-transform infrared spectroscopy (FTIR): JASCO FT-IR 4200 plus spectrophotometer with ATR (range 4000–400 cm^{-1} at 298 K).

Structural changes – powder x-ray diffraction (PXRD): Rigaku RINT 2500 X-ray diffractometer (CuKα anode; λ = 1.541 Ao). Samples scanned at 40kV and 30mA from 5^0 to 35^0 2θ values and analyzed using a PDXL2 software (Rigaku).

Configuration – transmission electron microscopy (TEM): High Resolution Transmission Electron Microscope (HR-TEM) Model of FEI –TECNAI G2-20 TWIN. Operating voltage 200kV. But for D-(+)-Glucose anhydrous: High resolution transmission electron microscopy (HR-TEM) on JEOL (JEM-2100 Plus) system under an acceleration voltage of 200 kV.

Method of spraying: MIRGA spraying is applied from 0.25 to 0.50 meter distance over the packaged laboratory chemicals. This distance is essential for the MIRGA sprayed solution to be able to form ion clouds, oscillation, and 2-6 µm mid-IR generation *(dealt in Discussion part)*. The mid-IR can penetrate the intervening package (polythene) and acts on the inside content (lab chemicals). Close spraying never generates energy.

Sensory profiling: The sensory attributes were tested by the sensory panel, which rated the samples using an acceptability index based on a hedonic scale with a 9-point nominal structure: Hedonic scale scoring used is: 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 [11, 12].

Sampling method in sodium bicarbonate: Two samples of sodium bicarbonate (50 gm) were packed in polyethene sachets. One of the samples was used as the non-sprayed control. The control sample was subjected to sensory analysis. The second sachet that served as the trial sample was subjected to one MIRGA spraying on one side from 0.25 to 0.50 meter distance. A portion of this sprayed sample was tested for a sensory evaluation. The packet was resealed and a second spraying on the other side was applied, sample taken, tested and resealed. Like this, the spraying, sampling and testing procedure was continued until the sample became unpalatable. For sodium bicarbonate, the fourth spraying enhanced palatability and the sixth spraying caused unpalatability, therefore the sprayings was stopped at six. The control, 4-sprayed and 6-sprayed sodium bicarbonate samples were subjected to instrumentation to identify the changes caused by MIRGA spraying.

Reason for more spraying: Spraying the chemical compound until losing its palatability was done because the input of an excess of energy to a target (lab chemical) should cause the denaturation of the target. It was expected that multiple MIRGA sprayings would denature the chemical compound.
The same method was applied individually to Sodium chloride and Anhydrous D-(+)-glucose samples, and results were compared. The sprayed chemicals with altered inherent characteristics were subjected to instrumentation tests.

Results and Discussion

Sensory Evaluation

Table 1: Sensory scoring of laboratory chemicals using 9-point hedonic scale

No of MIRGA spraying	Hedonic score		
	sodium bicarbonate *(Saltiness + hardness)*	**sodium chloride** *(Saltiness)*	**anhydrous D-(+)-glucose** *(Sweetness)*
0 (Control)	5	5	5
1	6	6	7
2	7	6	8
3	7	7	6
4	8	7	5
5	4	3	3
6	2	2	1

Sodium bicarbonate:

Control (non-sprayed) - Natural taste
4-sprayed sample - saltiness and hardness increased
6-sprayed sample - saltiness and hardness extremely reduced

Sodium chloride:

Control (non-sprayed) - Natural taste
4-sprayed sample - saltiness increased
6-sprayed sample - strong and unpleasant to taste

Anhydrous D-(+)-glucose:

Control (non-sprayed) - Natural sweetness
2-sprayed sample - sweetness increased
6-sprayed sample - sweetness reduced, chilling effect disappeared, tasted like sugar

Instrumentation results *(raw data of instrumentations and detailed interpretations present in Supplementary Data D1 and Text T2 respectively).*

(a) Sodium bicarbonate

GC-MS

Control sample contains 9-octadecenoic acid, derivative of hexahydropyridineas well as oleic acid as major peak. After 4 spraying, there was a new peak of 2-(acetoxymethyl)-3-(methoxycarbonyl) biphenylene, which is responsible for increase in saltiness. While 6-sprayed sample has shown novel peak of 2, 3-dihydroxypropyl elaidate as well as an increase of the peak of methyl hexadecanoate, which is responsible for reduction in saltiness. In addition, there were new peaks of diethyl phthalate.

FTIR

The broad signal in the range of 1600-1700 $cm^{-1}$is associated with the stretching vibration of C=O which is slightly increased in the 4-sprayed sample (saltiness and hardness increased) and further increased by around 7.5% in the 6-sprayed sample (saltiness and hardness extremely reduced). The observed changes in the C=O stretching vibration confirm the alterations occurred upon MIRGA spraying.

PXRD

PXRD patterns show that the samples are highly polycrystalline materials, with monoclinic P crystalline structures. Increase in relative intensities of signals at 2θ 18.4 to 127% (4-sprayed sample) and 106% (6-sprayed sample); 2θ 57.04 to 33% (4-sprayed sample) (saltiness and hardness increased) and 47% (6-sprayed sample) (saltiness and hardness extremely reduced), are evidence of an atomic re-arrangement in diffraction planes (020 and 060), due to sprayings, especially in the 4-sprayed sample.

TEM

The control sample shows morphology with whiskers (needle-like) of about 200 nm in length and clusters of crystals or individual crystallites of roughly 200 nm. Similarly, the 4-and 6-sprayed samples have needle like structures and crystallites. However, the below figure confirms the changes in elemental composition between the control and sprayed samples.

Sodium chloride

GC-MS

Control: Within the limit of noise, the GC-MS pattern shows peaks around 2.4 and 3.1 min with possible peaks around 6 min and 7 min.

4-sprayed sample: Compared to control, the resolution of this sample GC-MS pattern is improved. There are peaks at 3.4 min and 6 min (same as for control). There are distinct peaks at ~3.2 min, ~4 min, and ~7.9 min. These additional peaks are suggested to be relative to the increased saltiness of the 4-sprayed sample relative to the control sample.

6-sprayed sample: The GC-MS pattern is significantly different from that of control. The key differences are the missing peaks at 2.4 min, 3.1 min, 6 min, and 7 min. There are peaks at 7.9 min (same as for the 4-sprayed sample) and unique peaks at around 8.6 min, 10.4 min, 12 min, and 13.5 min. These differences are suggested to give rise to the strong and unpleasant taste of the sample.

FTIR

Differences in OH- stretching (at 3400 cm^{-1}) and H-O-H- deformation and combination bands (at 1640 cm^{-1} and 2340 cm^{-1}) are observed in the sprayed samples compared to control. There is an implication of these changes with the number of water molecules per salt molecules, and this could be the cause of the enhanced saltiness.

PXRD

Crystalline form of NaCl has peaked at 32º, 45.3º and 56º and more in 45.3º due to change in molecular coordinates and the change in salt particle's size due to MIRGA spraying.

TEM

TEM images clearly demonstrated the structural changes.

Control: Particle size (Feret's diameter) is below 1 µm for most observed particles. Mean and median values are both 313 nm (with 60% relative standard deviation).

4-sprayed sample: Particle size (Feret's diameter) is below 2 µm for most observed particles. Mean (median) values of particles below 1 µm and of particles with size in the range 1 – 2 µm are, respectively: 0.47 µm and 1.55 µm.

6-sprayed sample: This sample showed a significant change in morphology. Particle size statistics are not reliable, irregularly shaped, including holes, cluster with an aggregate of nanoparticles, with richer chemical composition in high Z element and the spherical particles are below 5µm in size.

Anhydrous D-(+)-glucose

GC-MS

Control: It shows apparent peaks at the following times (min): 16.8, 18.9, 19.2, and 20.9.

2 sprayed sample: It lacks the peaks observed in the control sample. These missing peaks may be related to the increased sweetness of this sample relative to the control sample.

*6-sprayed sample***:** It has a GC-MS pattern distinct from that of the control. There are key peaks distinct from the GC-MS of the control sample at 7.9 min, 9.8 min and 14.8 min. The four key peaks in the chromatogram of the control sample are not distinguishable from the noise in this sample. These major differences are ascribed to the reduced sweetness and change in properties of the 6-sprayed sample.

FTIR

The broad signal in the range of 3200-3600 cm^{-1}isassociated with the stretching vibration of O-H [13]. This signal increased by around 8% in 2-sprayed sample and drops by around 8.3% in the 6-sprayed sample, compared to the control. Furthermore, there is a signal appearing only in the 2-sprayed sample at around 1710 cm^{-1} associated with the stretching vibration of C=O [13] meaning that a chemical change occurred implying the creation of C=O bonds. This peak disappears again in the 6-sprayed sample.

PXRD

PXRD patterns show samples are highly polycrystalline materials, with orthorhombic structure typical of α-D anhydrous glucose, with maximum intensity at 2θ; 18.75 and additional peaks at 11.89; 14.62 and 17.05. There is a slight decrease in the intensities relative to 11.89, from 13% (control) to 8.6% (2-sprayed) and 8.2% (6-sprayed). This decrease is also observed in the peak at 17.05 but not in the maximum peak (18.75). These variations in intensity are due to slight imperfections of the crystals by effect of the MIRGA spraying.

TEM

With respect to the control, MIRGA spraying affect increasingly the following aspects:

Particle shape and state of aggregation: Regularly squared particles are only observed in the control, while in the 2-sprayed sample there are irregularities in the squared shape. In the 6-sprayed sample a complete loss of the squared shape. In parallel, particles are observed as individual objects in the control and the 2-sprayed samples, while in the 6-sprayed sample mostly aggregates of sub-micrometer and nanosized particles are observed (with few exceptions).

Quality of crystallinity and crystallites orientation: Increasing the number of sprays affects the reciprocal disposition of lattice planes within particles, disturbing the original arrangement of the control. The dislocation of lattice planes (Moirè patterns) likely causes the particles to lose their structural stability, with consequent rearrangement of crystallites, either within or outside particle body (chain-like clusters).

About MIRGA

Invention background, definition, technique of mid-IR generation from MIRGA, toxicological study on MIRGA, safety of the MIRGA sprayed usables and primeval and future scope of MIRGA have been described by Umakanthan *et al*., 2022a *(detailed discussion on MIRGA available in Supplementary Text T1).*

The 2-6 µm mid-IR applied over the chemicals caused –OH and C=O stretching, formation of new molecules, deformation of H–O–H, elemental changes and rearrangement of atoms which are responsible for the improved/ reduced characteristics of trialed laboratory chemicals depending on number of MIRGA sprayings applied. MIRGA technology rapidly influences the chemicals characteristics. Mid-IR has long wavelength and low frequency, and elicits vibrational modes in molecules through dipole movement changes [14-16], leading to the chemical bond parameter changes and formation of new molecules [17], causing an alteration in physico-chemistry [18,19] of laboratory chemicals, thus increasing / reducing their potency.

Action of MIRGA emitted 2-6 µm mid IR on the lab chemicals

While spraying MIRGA, most of the mid-IR energy scatters through the air and gets absorbed by receptors (edibles or usables) molecules. Virtually all organic compounds absorb mid-IR radiation which causes a change in molecule's vibrational state to move from the lower ground state to excited higher energy state [20]**.** This leads to changes in chemical bonds [21] and these bond parameter changes led to consequent changes in target's physical and chemical characters, configuration, compound transformation depending on the dose of energy applied [22-24]. Nanostructured water layers can be triggered upon application of mid-IR radiation, since water molecules absorb in this region. [25-26]

Depending on number of MIRGA spraying (energy given), a receptor's chemical bond configurations and subsequent physical and chemical characters can be altered to our desire.

As displayed in the results, 2-6 µm MIR generated from the MIRGA equipment caused chemical and molecular level changes in the chemical components. This action of the MIR is called as photodegradation which means alterations of materials by light. In this process, chemical components have absorbed the MIR generated by MIRGA spraying, and the absorbed MIR photons have broken their chemical bonds; thereby the molecules are degraded and transformed into another molecule/ compound, as reported in GCMS analysis.

We conducted different kinds of primary tests in lab and industries. The MIRGA irradiation caused 25-30% reduction in chemical (quantitative) requirements than the routine. The detailed study is ongoing and will be disclosed soon. This potentiating technology has possibility in the reduction of quantity requirement of other chemicals as well.

Similar desirable results in coffee, tea, cocoa, edible salts and terminalia were achieved using MIRGA spraying by Umakanthan *et al*., 2022a; Umakanthan *et al*., 2022b; Umakanthan *et al*., 2023c; Umakanthan *et al*., 2023d **[**1-4]**.**

Conclusions

Laboratory chemicals viz., sodium bicarbonate, sodium chloride and anhydrous D-glucose were irradiated with 2-6µm mid-IR. Comparing the non-irradiated control sample, the irradiated samples found to show physico-chemical changes, thereby enhanced/ reduced inherent characteristics including sensory attributes. This technology saves resources and economy.

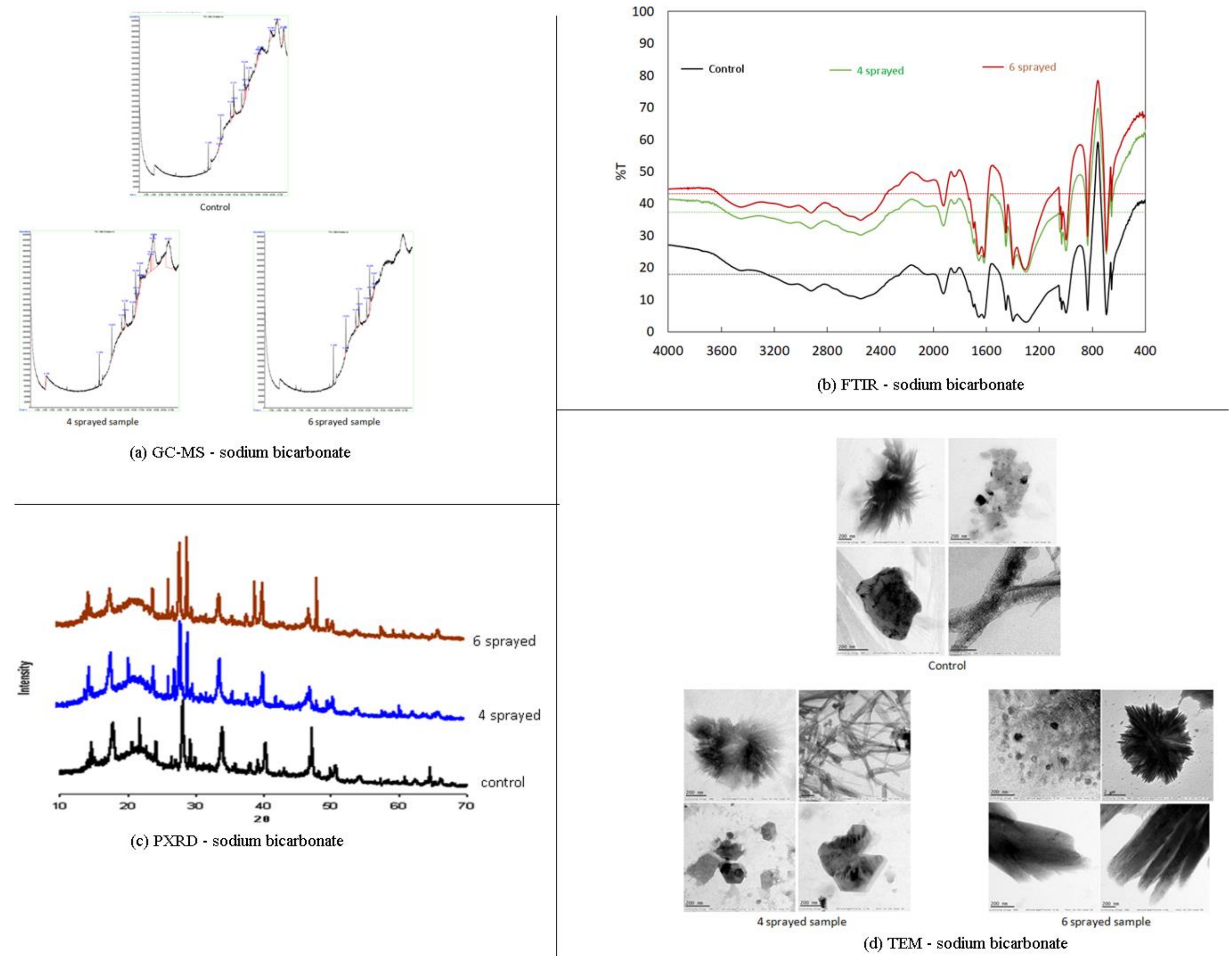

Figure 1: Instrumentations of Sodium bicarbonate samples
(a) GC-MS, (b) FTIR, (c) PXRD, (d) TEM

Spectrum: Control

Element	Series	Net unn.	C norm. [wt.%]	C Atom. [at.%]	C Error [wt.%]	(3 Sigma) [wt.%]
Carbon	K-series	7208	27.90	27.90	45.22	2.77
Oxygen	K-series	35201	19.46	19.46	23.67	1.85
Sodium	K-series	87831	26.62	26.62	22.54	2.49
Aluminium	K-series	3688	1.17	1.17	0.85	0.20
Copper	K-series	49361	24.35	24.35	7.46	2.29
Chlorine	K-series	1584	0.50	0.50	0.27	0.13
Total:			100.00	100.00	100.00	

Spectrum: 4 sprayed sample

Element	Series	Net unn.	C norm. [wt.%]	C Atom. [at.%]	C Error [wt.%]	(3 Sigma) [wt.%]
Oxygen	K-series	38461	15.43	15.43	15.22	1.48
Carbon	K-series	17632	49.53	49.53	65.11	4.67
Sodium	K-series	113742	25.02	25.02	17.18	2.34
Copper	K-series	27986	10.02	10.02	2.49	1.00
Total:			100.00	100.00	100.00	

Spectrum: 6 sprayed sample

Element	Series	Net unn.	C norm. [wt.%]	C Atom. [at.%]	C Error [wt.%]	(3 Sigma) [wt.%]
Oxygen	K-series	69769	24.90	24.90	23.23	2.33
Carbon	K-series	20526	51.30	51.30	63.76	4.82
Sodium	K-series	91509	17.91	17.91	11.63	1.70
Copper	K-series	18492	5.89	5.89	1.38	0.62
Total:			100.00	100.00	100.00	

Figure 2: HR-TEM of sodium bicarbonate samples – Elemental analysis

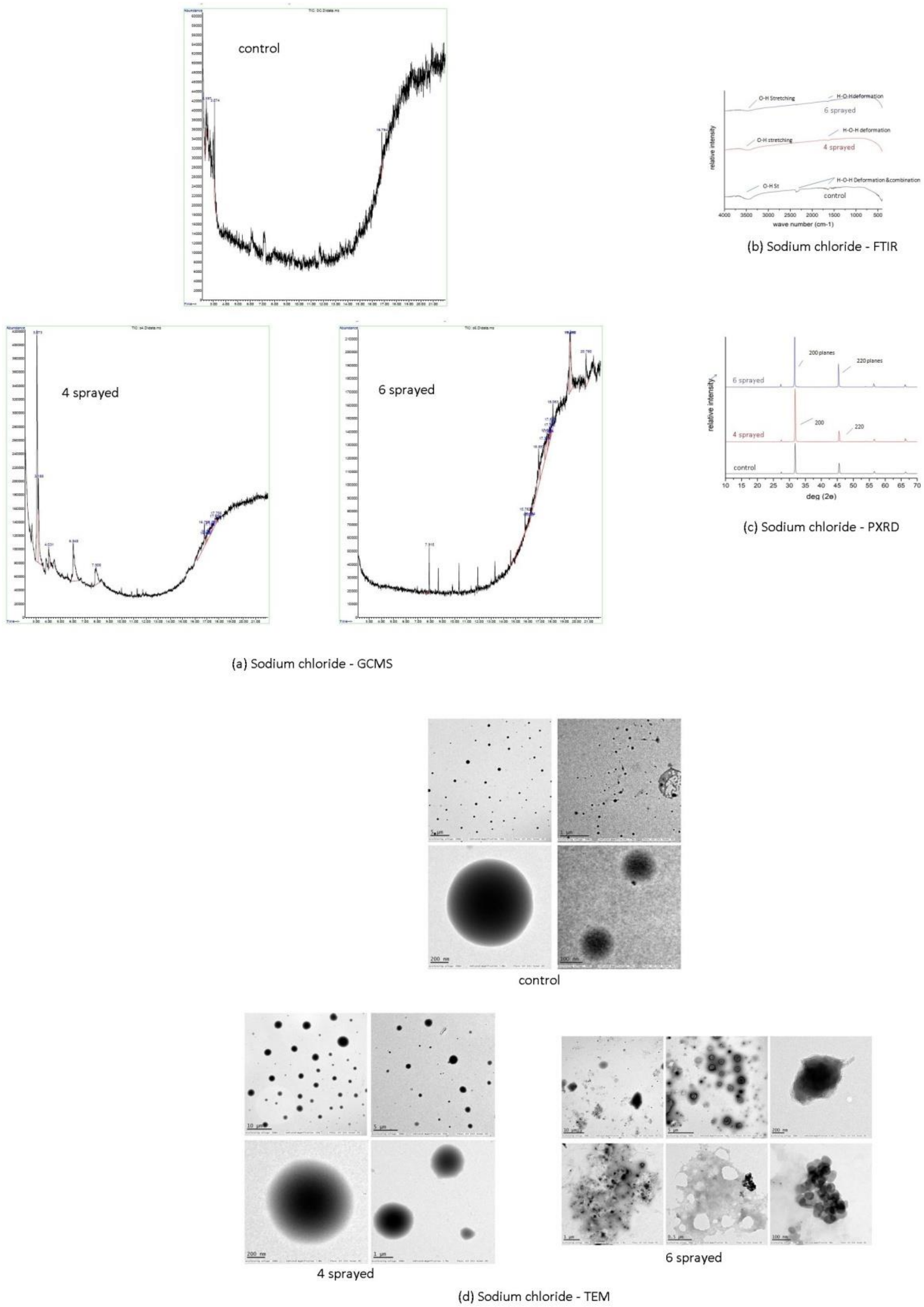

Figure 3: Instrumentations of Sodium chloride samples (a) GC-MS, (b) FTIR, (c) PXRD, (d) TEMGCMS

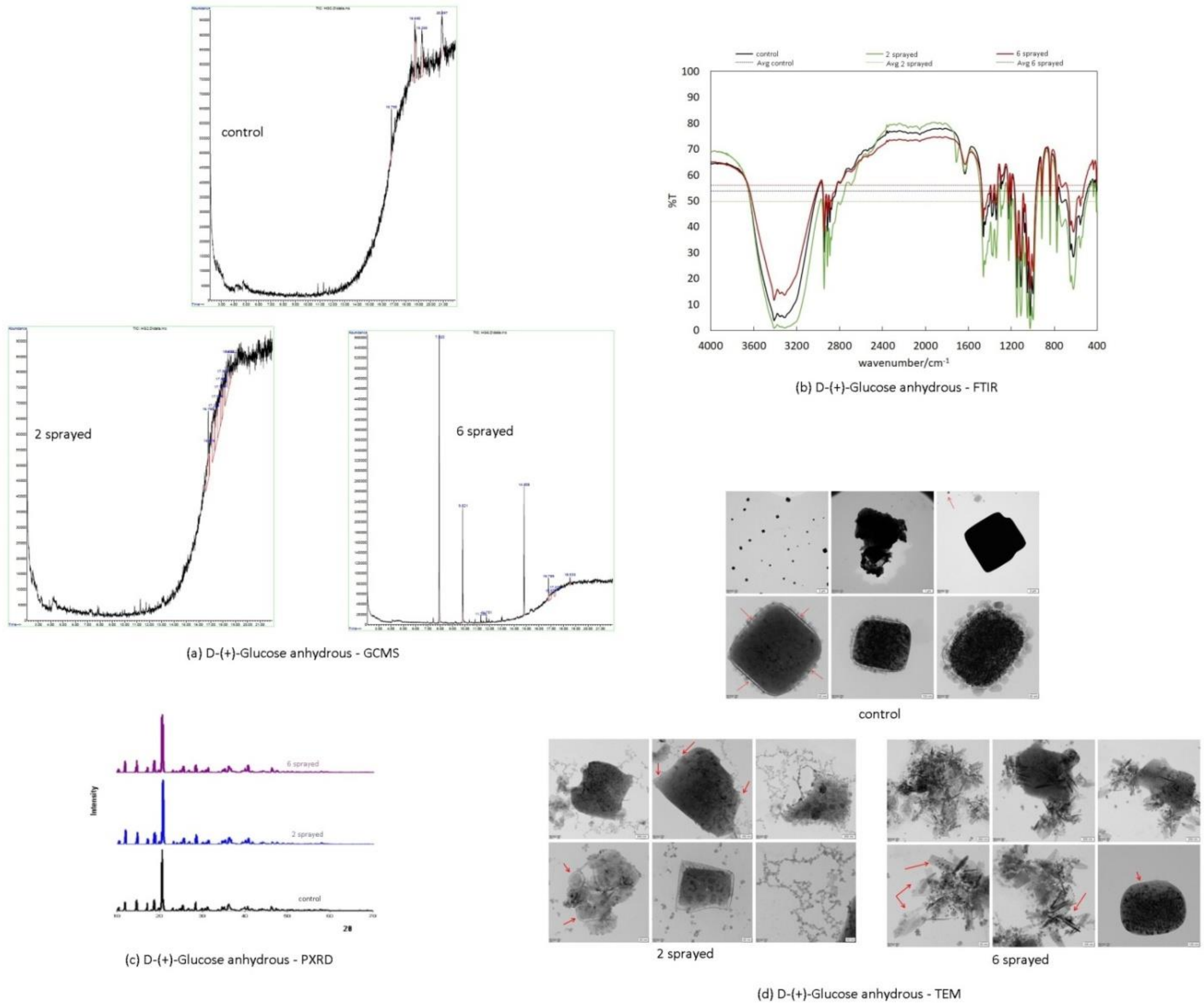

Figure 4: Instrumentations of Anhydrous d-(+)-glucose samples (a) GC-MS, (b) FTIR, (c) PXRD, (d) TEM

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Author contribution

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

Competing 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.

Data and materials availability

All data is available in the manuscript and supplementary materials.
Supplementary file: https://docs.google.com/document/d/1D-RiEWeumHgJc29TUH46IYXIR-ryQND2/edit?usp=sharing&ouid=111101387151809704391&rtpof=true&sd=true

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