Synthesis of Nano curcumin Via Sol-Gel / Ultrasonic Processors Route and Improving their properties by Microwaves-Induced Plasma

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Abstract Turmeric or Curcuma is a natural creation, whose therapeutic properties have been widely studied and an extensive variety of therapeutic effects on several diseases. Nano curcumin particles were prepared by Vibra-Cell™ Ultrasonic Liquid Processors device. The particles are characterized by scanning probe microscope (SPM) and Ultraviolet-visible spectroscopy (UV-Vis spectrophotometer). The results confirm that the prepared Nano curcumin has mean diameter 82 nm. The prepared Nano curcumin is exposed to plasma to enhance the properties of their Nano particle the result is improve the enhanced characterization.

Keywords. Nano-curcumine, curcumin, cold plasma, scanning probe microscope, UV-Vis spectrophotometer.

1. Introduction
Curcumin it is molecular formula C₂₁H₂₀O₆) has also known as (curcuma longa) or diferuloylmethane and it first known by Milobedeska and Lampe in 1910[1]. Curcumin a natural element, a polyphenolic compound extracted from the rhizome of the spice plant Curcuma longa, turmeric is yellowish in color, this because it contained curcumin, curcumin has been used extensively for treatment in traditional Chinese medicine and Ayurvedic. Curcumin possesses anti-oxidative, antidepressant, neuroprotective actions and anti-inflammatory and acts through several intracellular mechanisms affecting multiple targets. Curcumin has been proved to be effective for the treatment of different tumor, Alzheimer’s disease asthma, allergic reactions, and pathological disorders, such as rheumatoid arthritis and inflammatory bowel disease [2]. Curcumin has also been importance because of its effectiveness as a chemo-preventative agent, and also it is a chemo-/radio-sensitizer for tumorous cells, while acts as a chemo-/radio-protector for normal functioning organs [3, 4, 5]. However, poor solubility in water and inadequate oral bioavailability has been the reasons for its restricted use in clinical trials [3]. Turmeric contains 5.1% fat, 6.3% protein, 69.4% carbohydrates, 5.8% essential oils, and 366% of curcuminoïd; the main curcuminoïds in commercial curcumin are77% curcumin (curcumin I), z17% demethoxycurcumin (curcumin II), z3% bis-demethoxycurcumin (curcumin III), and cyclocurcumin (curcumin IV) [4]. Curcumin is a hydrophobic, polyphenolic compound, hence insoluble in water at acidic and neutral pH conditions, however soluble in ethanol, acetone, dimethylsulfoxide, methanol [1].

Figure (1) Shows the chemical structure and Turmeric (a) the curcumin Crystallized powder (b) the turmeric root. (c) The keto forms and enol of curcumin. The enol form is more dynamically stable in the solution and in solid phase [5, 8].

For enhancement the properties of the Nano curcumin it is exposed to plasma. Plasma is gas in which there is partial or full ionization such that it consists of positive and negative ions of approximately equal numbers. It has two main types (i.e. thermal plasma and non-thermal plasma). Non-thermal or microwave plasma which is used in this paper is produced from microwave sources in the GHz range without the need for electrodes [9, 10, 11, 12].
2. Experimental method

100 g of turmeric, volume weight mean 173.252 μm, surface weighted mean 60.021 μm, size range 0.020-2000.000 μm, 1000 ml of distilled water, Vibra -Cell Ultrasonic Liquid Processors device 750 Watt and 20 kHz. Ultrasonic Processors is a wide range of inorganic and organic materials, from 250 microliters to 1 liter ideal use include nanotechnology (producing nanoparticle materials and Graphene dispersions), sample preparation, homogenization, cell lysing, ChIP Assay, emulsification, deagglomeration and disaggregation, plus uses in the field of sonochemical processing.

2.1. Preparation of Nano-curcumine

To prepare the 100g Nano-curcumine, crushed curcumin is added to 1000 ml of distilled water then the mixture is melted on a low heat and the mixture is dissolved to get rid of the sediment. The solution is distributed in dishes and left to dry for two days, after two days powder is collected from dishes and the collected powder weight is 20 g of dried turmeric, then Add 800 ml of distilled water and expose the mixture to the Vibra-Cell Ultrasonic Liquid a device for an hour and a half where the device works 10 seconds and rest for 5 seconds. This procedure continuous 10 days. Finally Nano-curcumin is obtained.

3. RESULTS AND DISCUSSIONS

The results show that turmeric is converted into a Nano curcumin with particle size 82 nm. The Nano particles re improved by exposure to the microwave plasma for 25 min. The exposure to microwave plasma enhances the properties and reduced particle size to 47, which meant that the cold plasma effect is positive on Nano Curcumin it makes the particle more homogenous and tend to put them in a parallel form. The Roughness Average is \(22.1\) nm before expose to plasma and become \(0.459\) nm which gives good result in obtaining the nano particle, the Root Mean Square, Surface Skewness, Surface Kurtosis, Peak-Peak and Ten Poi Height, all these features become less in expose to microwave plasma.

3.1. Characterization of curcumin

To characterize the particle of curcumin, three types of analysis are used to improve that: particle size analyzer measurement by (MASTERSIZER 2000) where the results shows that the volume weight mean 173.252 μm, surface weighted mean 60.021 μm, size range from( 2-2000.000 μm ) figure (2) shows the particle size and its volume. the UV-VIS spectrophotometer for curcumin shows the wave is 422nm. X-ray diffraction (XRD) X-ray diffraction is the method to determine crystalline structure or phase of crystal and the X-ray diffraction pattern of synthesized is shown in figure (3).
3.2. Characterization of Nano-curcumin

To characterize the particle two type of analysis is used to shown the Nano-curcumine that is prepared. (SPM) Scanning probe microscope is a tool used for studying surfaces at the level in nanoscale. SPM use a physical probe to scan forth and back over all the surface of a sample and collect data, usually got as a two-dimensional grid of data points and displayed as a computer image during this scanning process, a computer collect data that are used to produce an image of the surface. SPM used to study the characterized of the nanoparticle and it shows according to table (1) the average diameter is 82.80 nm and there is 10% the diameter less than 65.00 nm and 50% the diameter is less than 80.00 nm and 90% is less than 95.00 nm. Table (2) represents the distribution of nanoparticle and its volume for each diameter is from (60nm-105nm) the volume and accumulation for particle. While Figure (4) represents the distribution of particle, it shows the homogeneity structure for nanoparticle. It shows how the particle line up following to each other in a way regularly and visibly.

| Diameter(nm) | Volume (%) | Cumulation(%) |
|--------------|------------|---------------|
| 60.00        | 2.05       | 2.05          |
| 65.00        | 5.13       | 7.18          |
| 70.00        | 7.69       | 14.87         |
| 75.00        | 12.31      | 27.18         |
| 80.00        | 11.28      | 38.46         |
| 85.00        | 15.90      | 54.36         |
| 90.00        | 14.87      | 69.23         |
| 95.00        | 15.38      | 84.62         |
| 100.00       | 11.79      | 96.41         |
| 105.00       | 3.59       | 100.00        |
Figure 4. Reveals the distribution of particle and Homogenous structure.

Figure (5) the reveals distribution of particle and the scale show that the particle size from 0 nm to 100.00 nm according to the color (black to white) is distributed. Some parameter is shown by CSPM imager Surface Roughness Analysis Image and these parameter is.

Figure 5. Reveals the distribution of particles and the scale shows that the particle size from 0 nm to 100.00 nm according to the color (black to white) is distributed.

Some parameters shows the Surface Roughness Analysis Image and this parameter are mentioned in Table (3):
3.3. Ultraviolet-visible spectroscopy (UV-Vis spectrophotometer)
UV-Visible spectroscopy is a very important experimental technique for measuring the optical properties of the sample. It has the ability to pass into the sample and to transmit through it. Transmittance (T), absorbance (A) and reflectance (R), absorbance is given by the relation:

\[ A = \log \left( \frac{1}{T} \right) \]  

The spectrum obtained is the absorbance one show a different wavelength in its compound the chemical structure of the molecule. Wavelength scanning by UV-VIS spectrophotometer showed absorption maximum at 423 nm. This is illustrated in figure (6).

![Figure 6](image)

3.4. Enhance the properties of nanoparticles
The Nano curcumin exposed to plasma for 25 min. Using microwaves-induced plasma system to generate non-thermal atmospheric pressure, plasma which operated with frequency 2.45 (GHz), microwave power of 800 W and utilized argon gas to generate the torch. Scanning probe microscope is used to show the effect of plasma on particle size. The results show that the average Diameter becomes

Table 3. Surface Roughness parameter method one

| Parameter | Value       |
|-----------|-------------|
| Sa (Roughness Average) | 23.1 nm     |
| Sq (Root Mean Square) | 26.1 nm     |
| Sk (Surface Skewness) | - 0.25      |
| Sm (Surface Kurtosis) | 2.08         |
| Sd (Peak-Peak) | 104 nm       |
| Sa (Ten Point Height) | 58.3 nm     |

... (continued table)
47.92 nm. This means that the average Diameter decreases after exposure to plasma by about 34.88 nm. one can be concluding that the plasma enhances the properties of Nano particle by reduced the size of particle. Table (4) shows the range of particle size.

Table 4. The diameter of the Nano curcumin after exposed to plasma for 25 min.

| Diameter (nm) | Volume (%) | Accumulation (%) |
|--------------|------------|------------------|
| 30.00        | 1.53       | 1.53             |
| 32.00        | 2.68       | 4.21             |
| 34.00        | 4.21       | 8.43             |
| 36.00        | 8.81       | 17.24            |
| 38.00        | 4.60       | 21.84            |
| 40.00        | 3.45       | 25.29            |
| 42.00        | 8.05       | 33.33            |
| 44.00        | 5.36       | 38.70            |
| 46.00        | 4.60       | 43.30            |
| 48.00        | 9.20       | 52.49            |
| 50.00        | 6.51       | 59.00            |
| 52.00        | 5.36       | 64.37            |
| 54.00        | 3.83       | 68.20            |
| 56.00        | 5.75       | 73.95            |
| 58.00        | 3.83       | 77.78            |
| 60.00        | 6.13       | 83.91            |
| 62.00        | 4.98       | 88.89            |
| 64.00        | 5.75       | 94.64            |
| 66.00        | 3.07       | 97.70            |
| 68.00        | 2.30       | 100.00           |

Table 5. The distribution, accumulation and volume of Nano-particles after plasma.

| Diameter (nm) | Volume (%) | Accumulation (%) |
|--------------|------------|------------------|
| 30.00        | 1.53       | 1.53             |
| 32.00        | 2.68       | 4.21             |
| 34.00        | 4.21       | 8.43             |
| 36.00        | 8.81       | 17.24            |
| 38.00        | 4.60       | 21.84            |
| 40.00        | 3.45       | 25.29            |
| 42.00        | 8.05       | 33.33            |
| 44.00        | 5.36       | 38.70            |
| 46.00        | 4.60       | 43.30            |
| 48.00        | 9.20       | 52.49            |
| 50.00        | 6.51       | 59.00            |
| 52.00        | 5.36       | 64.37            |
| 54.00        | 3.83       | 68.20            |
| 56.00        | 5.75       | 73.95            |
| 58.00        | 3.83       | 77.78            |
| 60.00        | 6.13       | 83.91            |
| 62.00        | 4.98       | 88.89            |
| 64.00        | 5.75       | 94.64            |
| 66.00        | 3.07       | 97.70            |
| 68.00        | 2.30       | 100.00           |

From Figure (7) it's observed that the distribution of nanoparticles is regular and homogeneous after exposing to plasma. Figure (8) shows the particle structure distribution. Figure (8), according the scale, shows that the particle size from 0 nm to 100.00 nm according to the color (black to white) is distributed and some parameters which can be obtained are listed in Table (6).

Figure 7. The distribution of particle and Homogenous structure after plasma.
Figure 8. The distribution of particle and the scale show that the particle size from 0 nm to 100.00 nm according to the color (black to white) is distributed.

Table 6. SPM parameter after plasma method one

| Amplitude parameters | \( S_a \) (Roughness Average) | 0.459 nm |
|----------------------|------------------------------|----------|
| 2                    | \( S_{sq} \) (Root Mean Square) | 0.529 nm |
| 3                    | \( S_{sk} \) (Surface Skewness) | 0.288 nm |
| 4                    | \( S_{ku} \) (Surface Kurtosis) | 1.86     |
| 5                    | \( S_{pk} \) (Peak-Peak) | 1.9 nm |
| 6                    | \( S_{z_T} \) (Ten Point Height) | 1.9 nm |

| Hybrid Parameters | \( S_{sc} \) (Mean Summit Curvature) | 0.00229(1/nm) |
|------------------|--------------------------------------|----------------|
| 2                | \( S_{skc} \) (Root Mean Square Slope) | 0.0305(1/nm)  |
| 3                | \( S_{sr} \) (Surface Area Ratio) | 0.0456         |

| Functional parameters | \( S_{sb} \) (Surface Bearing Index) | 3.17 |
|-----------------------|--------------------------------------|------|
| 2                    | \( S_{cf} \) (Core Fluid Retention index) | 1.65 |
| 3                    | \( S_{v} \) (Valley Retention index) | 0.0512 |
| 4                    | \( S_{sh} \) (Reduced Summit Height) | 0.583 nm |
| 5                    | \( S_{cr} \) (Core Roughness Depth) | 1.19 nm |
| 6                    | \( S_{rv} \) (Reduced Valley Depth) | 0.088 nm |
| 7                    | \( S_{dc} \) (0-5 % height intervals of Bearing Curve) | 0.167(1/nm) |
| 8                    | \( S_{dc} \) (5-10 % height intervals of Bearing Curve) | 0.113 (1/nm) |
| 9                    | \( S_{dc} \) (10-50 % height intervals of Bearing Curve) | 0.859 (1/nm) |
| 10                   | \( S_{dc} \) (50-95 % height intervals of Bearing Curve) | 0.64 (1/nm) |

| spatial Parameters | \( S_{sd} \) (Density of Summits) | 266(1/\( \mu \text{m}^2 \)) |
|-------------------|----------------------------------|-----------------------------|
| 2                 | Fractal dimension | 2.47 |
The UV-VIS spectrophotometer showed absorption maximum at 422 nm. Analysis of the data means that the material has kept its spectral properties constant and has not changed after exposure to plasma. That confirmed the presence of curcumin (figure 9).

4. CONCLUSION
Nano-curcumin was manufactured in a simple and inexpensive way. The results showed that particles are nanoparticles and can be used in industrial, medical fields and in the treatment of diseases. The microwave plasma can improve the properties of nanoparticles and that showed through the above tables and result. The UV spectrophotometer test shows the same curve before and after exposure to plasma, this means that the material has kept in its spectral properties.

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