Novel core-shell bioceramic silica-Berberis vulgaris: synthesis and characterization

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ABSTRACT
Enhancements in bioceramic mixtures represent a significant avenue for achieving superior mechanical and biological properties. Therefore, the present study aimed to extract active compounds from Berberis vulgaris stems and fruits collected from the Khorasan province, employing advanced analytical techniques such as GC-MS and FTIR to elucidate the composition of these extracts. The derived extracts were utilized to synthesize novel nanocomposites, denoted as SiO$_2$-MPS-stem extract and SiO$_2$-MPS-fruit extract. Comprehensive characterization of these composites was conducted through SEM, EDX mapping, FTIR, and XRD analyses. The characterization measurements validated the successful coating of silica with the extracts, resulting in a core-shell nanostructure with particle sizes below 60 nm. These composites were incorporated into bioceramics for dental root fillings with an equal weight ratio. The bioceramic material was subjected to the same aforementioned characterization techniques, revealing that their sizes fell within the nanoscale range, not exceeding 70 nanometers. The results indicated a core-shell configuration for the nanomaterials, with the shell comprising the bioceramic component of bioceramic-SiO$_2$-MPS-fruit extract and bioceramic-SiO$_2$-MPS-stem extract.

Introduction

The root canal treatment procedure involves using endodontic files to widen the root canal and applying chemical disinfectants to cleanse the area. The process aims to eliminate any remaining living or dead tissues, eradicate the microbes residing in the root canal system, disrupt any microbial biofilm, and eliminate any accumulated hard debris resulting from the root canal instrumentation. After that, packing and sealing the canal with a suitable sealing material to achieve a fluid-tight seal space in the channel (1).

Endodontics is an evolving field driven by continuously introducing new technologies and techniques. The significant growth in endodontics is mainly attributable to advancements in endodontic material sciences. One recent groundbreaking material that has transformed endodontics is the introduction of bio-ceramic root canal obturation sealers (2).

Calcium silicate-based bio-ceramic endodontic sealers (Portland Cement) have many remarkable properties for good prognosis of the endodontic treatment; their resemblance to biological hydroxyapatite gives them outstanding biocompatibility properties. They also possess intrinsic osteoinductive capabilities that enable them to absorb osteoinductive substances in cases where bone healing occurs nearby. In addition, they serve as a regenerative scaffold of resorbable lattices that offer a framework for tissue rebuilding, which eventually dissolves as the body undergoes tissue regeneration (3). The eradication of bacteria from the root canal involves chemical disinfection and mechanical Preparation of the canal system. However, eliminating microbes from the canal system is unachievable despite the available chemical irrigation and automated strategies. Therefore, using root canal-filling materials that possess antibacterial properties is advantageous. Calcium silicate-based bio-ceramic endodontic sealers have excellent antibacterial properties; bacterial sequestration occurs due to precipitation in situ after setting, resulting in antibacterial properties, especially against Enterococcus faecalis (4). Due to the increasing resistance of pathogenic bacteria to antibiotics and chemotherapeutic agents, researchers are increasingly focused on exploring alternative products and treatment options for oral diseases.

Consequently, natural phytochemicals extracted from plants and traditionally used in medicine are emerging as viable replacements for synthetic chemicals (5). Berberis vulgaris member of the Berberidaceae family thrives across Asia and Europe. The B. vulgaris is a 1–3 m tall shrub with prickly yellow bark and oblique leaves, followed by yellow blooms and round red berries. This plant's roots, bark, leaves, stem, and fruit have all been used for medicinal purposes. This plant's primary alkaloids are berberine, berberine, and palmitine. Its principal constituents provide diverse antibacterial effects (6).

Another attempt to enhance the endodontic sealing material is the incorporation of nanomaterials into the endodontic sealing materials; nanotechnology refers to the investigation of biology, technology, and architecture at microscopic scales, typically ranging from 1 to 100 nanometers. Its applications span various sectors and involve manipulating the size and structure of nanoparticles at a scale of one billionth of a meter. In simpler terms, nanotechnology involves creating functional entities that are 100 nanometers or smaller in size (7). The utilization of plants for the synthesis of nanoparticles (NPs) is an ad-

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Materials and Methods

The materials used in this study were obtained from Sigma-Aldrich. The Berberis vulgaris stems and fruits were collected and processed in-house. Analytical grade solvents and reagents were employed for extraction and synthesis processes; the following materials were used:
1. Berberis vulgaris stems: Fresh stems of Berberis vulgaris were collected and carefully selected for the extraction of active compounds.
2. Berberis vulgaris fruits: Fresh fruits of Berberis vulgaris were collected and prepared for the extraction of active compounds.
3. Solvents: Analytical grade solvents (deionized water, methanol, cyclohexane) were obtained to extract the active compounds from the Berberis vulgaris stems and fruits and to prepare the nanocomposites.
4. γ-methacryloxypropyltrimethoxysilane (MPS) was utilized for the functionalization of the silica.
5. Bioceramic sealer was purchased from south korea.

Instruments


Berberis extraction procedure

To extract the medicinal compounds from the Berberis plant, the stems and fruits were processed separately using a series of steps.

Firstly, the plant parts were carefully placed in a shaded area to dry out completely. Once dried, the plant parts were finely ground into a powder using a blender.

In detail, the powdered plant material was mixed with 70% ethyl alcohol, maintaining a ratio of 1 part plant powder (36.2935g) to 7 parts alcohol (254 ml). The mixture was left to stir at 750 rpm at boiling temperature for 48 hours in the Clevenger apparatus, allowing the alcohol to extract the desired compounds effectively.

Following the extraction period, the resulting mixture underwent filtration twice using Whatman No. 4. Subsequently, the extracts were centrifugated at -4 °C for 10 minutes at 11745 rpm to separate any remaining impurities.

To eliminate any residual traces of ethanol and water, specific measures were taken. The extracts were placed in an incubator packed with calcium chloride for ten days at a controlled temperature of 37 °C to evaporate the ethanol content; lastly, the extracts obtained from different parts of the Berberis plant were subjected to sterilization using 0.22 μm micropore filters, ensuring the removal of any potential contaminants.

Silanization of silica nanoparticles

In this experimental procedure, a synthesis route for obtaining 3-methacryloxypropyltrimethoxysilane- (γ-MPS-) silanized SiO₂ nanoparticles were employed. Initially, a mixture comprising 5.0 g of SiO₂ nanopowder, 0.1 g of n-propylamine, and 0.6 g of γ-MPS was introduced into a beaker containing 100 mL of cyclohexane. The components were subjected to ultrasound (probe 6, 150 Watt) for 2 minutes, and then the mixture was stirred at room temperature for 30 minutes to ensure thorough dispersion. Subsequently, the suspension underwent continuous stirring for an additional 30 minutes while the temperature was maintained at 65 °C — this controlled thermal treatment aimed to promote the reaction kinetics and enhance the desired functionalization process. Following the reaction period, the solvent was removed utilizing a rotary evaporator set at 65 °C, facilitating the evaporation process. The resulting precipitates were then subjected to a series of washing steps, using absolute ethanol as the washing agent. This rigorous washing procedure was performed three times to eliminate any residual unreacted γ-MPS and ensure the purity of the obtained functionalized nanoparticles. To finalize the synthesis, the washed powders were subjected to vacuum drying at 80 °C for 24 hours. This drying step aimed to remove any remaining solvent and facilitate the formation of a dry, stable product suitable for subsequent Characterization and analysis.

Synthesis and characterization of berberis extract–silica nanoparticle

The Silanization of berberis stem or berberis fruit extract was carried out using the batch method. The loading ratio employed was m(SiO₂):m(berberis)= 10:1. To summarize the procedure, SiO₂ and berberis extracts were dissolved in a solution of polyethylene glycol-400 (1% in distilled water) and stirred for 24 hours at 30 °C. Subsequently, the solution underwent centrifugation (SSU-173) at 12000 rpm for ten minutes. The resulting material, loaded with the berberis extract, was then subjected to repeated washing with distilled water and subsequently dried at a temperature of 50 °C overnight, yielding the final product: SiO₂-MPS-Fruit extract and SiO₂-MPS-Stem extract nanocomposites.

Preparation of the dental sealer composites

The specific amounts of SiO₂-MPS extracts (1%, 2%, and 3%) were carefully measured and combined with the bioceramic sealer paste. Subsequently, the mixture underwent a rigorous agitation process, employing a magnetic stirrer and a mechanical stirrer (dental stone laboratory engine) for 20 minutes. This dual agitation method ensured comprehensive mixing and dispersion of the SiO₂-MPS extracts within the bioceramic sealer paste, promoting even distribution of the constituents. The combined action of these stirring techniques facilitated the formation of a uniform mixture, thereby ensuring optimal integration of
tion of the Si–O–Si bond (symmetrical), which is another characteristic feature of silica.

6. The band at 470 cm\(^{-1}\) is due to the bending vibration of the Si–O–Si bond (asymmetrical).

The presence of these bands is an indication of the existence of silica in its pure amorphous form (11, 12, 13).

**XRD**

Silica nanoparticles exhibit broad XRD peaks due to their small size, resulting in decreased diffraction intensity and peak broadening. The peaks observed in the XRD pattern of the provided silica nanoparticles occur at approximately 21.8° and 33.4° (2θ). These correspond to the (101) and (200) crystal planes of hexagonal silica, respectively (14, 15). The d-spacing for these peaks is 4.08 Å and 2.68 Å, respectively. The full width at half maximum (FWHM) of the peaks indicates the degree of crystallinity, which in this case is 0.87°2θ for both peaks. See (Figure 1 and Table 1).

**SEM and EDX of SiO\(_2\) nanoparticles**

The SEM characterization of the silica nanoparticles in (Figure 2 and Table 2) shows that the particles have a spherical morphology and are well-dispersed in the sample.

**Results**

**Characterization of SiO\(_2\) nanoparticles and its nano-composite**

**SiO\(_2\) nanoparticles**

**FTIR**

In the case of silica nanoparticles, the FTIR spectrum (Figure S1 and Table S1) can provide insight into the chemical composition and structure of the material. The prominent peaks observed in the FTIR spectrum of silica nanoparticles are:

1. The band at 3410 cm\(^{-1}\) is due to the stretching vibration of the hydroxyl (–OH) group, which is typically present on the surface of silica nanoparticles.
2. The band at 1654 cm\(^{-1}\) is due to the bending vibration of the –OH group, which is also present on the surface of the nanoparticles.
3. The band at 1095 cm\(^{-1}\) is due to the stretching vibration of the Si–O–Si bond (asymmetrical), a characteristic silica feature.
4. The band at 956 cm\(^{-1}\) is due to the stretching vibration of the Si–O–H bond, which is also present on the surface of the nanoparticles.
5. The band at 806 cm\(^{-1}\) is due to the bending vibration of the Si–O–Si bond (symmetrical), which is another characteristic feature of silica.

The presence of these bands is an indication of the existence of silica in its pure amorphous form (11, 12, 13).

**Table 1. XRD data and crystalline size of SiO\(_2\) derivative.**

<table>
<thead>
<tr>
<th></th>
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<tbody>
<tr>
<td>1</td>
<td>21.7877</td>
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<td>10.6 Mean (10.7)</td>
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<td></td>
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<tr>
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<td>4.30488</td>
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<td>Fruit-MPS</td>
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<td>Stem-MPS</td>
<td>22.5974</td>
<td>4.31000</td>
<td>0.9001</td>
<td>9.40</td>
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</tbody>
</table>

**Scheme 1. Synthesis step of the bioceramic composites.**

![Scheme 1. Synthesis step of the bioceramic composites.](image)

the SiO\(_2\)-MPS extracts with the bioceramic sealer paste, as illustrated in the scheme (1).

![Figure 1. XRD of SiO\(_2\) NPs.](image)

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**Table 2. SEM data of silica nanoparticles.**

<table>
<thead>
<tr>
<th>Characteristic</th>
<th>Observations</th>
</tr>
</thead>
<tbody>
<tr>
<td>Morphology</td>
<td>Spherical</td>
</tr>
<tr>
<td>Particle Size Range</td>
<td>15.55-29.34 nm</td>
</tr>
<tr>
<td>Particle Dispersion</td>
<td>Well-dispersed</td>
</tr>
</tbody>
</table>

**Table 2. SEM data of silica nanoparticles.**

![Figure 1. XRD of SiO\(_2\) NPs.](image)
The average particle sizes are 15.55-29.34 nm, indicating that the model contains a range of particle sizes. (16, 17). The EDX analysis of the silica nanoparticles in (Figure S2) shows two peaks of Si and O at 1.74 and 0.52 keV. Furthermore, the EDX offers that the particles are composed mainly of oxygen (50.5 wt%) and silicon (49.5 wt%). This elemental composition is consistent with the design of silica (O, 53.26; Si, 46.74), which has a chemical formula of SiO₂. The well-dispersed nature of the nanoparticles is also observed in the EDX mapping, which shows a homogeneous distribution of oxygen and silicon across the sample. The EDX analysis provides essential information about the elemental composition of the nanoparticles.

**MPS nanoparticles**

**FTIR**

The FTIR spectrum of SiO₂ functionalized with 3-methacryloxypropyltrimethoxysilane reveals characteristic bands as illustrated in (Figure S3 and Table S2). The band at 3433 cm⁻¹ indicates hydroxyl (-OH) groups on the nanoparticle surface, implying hydrophilicity and potential interaction with polar molecules. The band at 2954 cm⁻¹ represents the methylene (-CH₂-) groups of the coupling agent, confirming successful attachment. The band at 1720 cm⁻¹ corresponds to the carbonyl (C=O) group, verifying the presence of the coupling agent. The band at 1630 cm⁻¹ confirms the methacrylate functional group. Bands at 1459 cm⁻¹, 1435 cm⁻¹, 1099 cm⁻¹, 798 cm⁻¹, and 470 cm⁻¹ provide further evidence of the SiO₂ core in the functionalized nanoparticles (18, 19).

**XRD**

The XRD pattern of the silanized silica nanoparticles (Figure 3) shows one prominent peak at approximately 22.6° (2θ). This peak corresponds to the (101) plane of the hexagonal crystal structure of silica, with a d-spacing of 4.30 Å. The peak's full width at half maximum (FWHM) is 0.92°2θ, indicating that the silanized silica nanoparticles are relatively amorphous with some degree of crystallinity. The crystallite size can be estimated using the Scherrer equation, which relates the FWHM to the crystallite size. Based on the FWHM and assuming a spherical crystallite shape, the crystallite size is estimated at 9.21 nm. This value suggests that the silanized silica nanoparticles have a relatively small crystallite size, consistent with the small particle size of silica nanoparticles. It should be noted that the XRD pattern provided only shows one peak, which indicates that the silanized silica nanoparticles are not highly crystalline. This could be due to the modification of the surface properties of the nanoparticles through Silanization, which may have disrupted the crystal structure of the nanoparticles (19).

**SEM and EDX of mesoporous silica nanoparticles**

The SEM analysis indicates that after functionalizing with 3-methacryloxypropyltrimethoxysilane, the particle size range increased to 17-36.43 nm, with some changes in the structure. This change in size and layout is likely due to the addition of the functional group, which can lead to aggregation or rearrangement of the nanoparticles. However, the particles still maintain a spherical shape and are well dispersed, indicating that the functionalization process did not significantly affect the overall morphology or dispersion of the nanoparticles. See (Figure 4 and Table 3).

The EDX analysis in (Figure S4) indicates that the SiO₂ nanoparticles have been successfully functionalized with 3-methacryloxypropyltrimethoxysilane, as evidenced by the presence of carbon (C) in addition to the expected silicon (Si) and oxygen (O) elements. The weight

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**Table 3. SEM data with SiO₂-3-methacryloxypropyltrimethoxysilane.**

<table>
<thead>
<tr>
<th>Characteristic</th>
<th>Observations</th>
</tr>
</thead>
<tbody>
<tr>
<td>Morphology</td>
<td>Spherical</td>
</tr>
<tr>
<td>Particle Size Range</td>
<td>17-36.43 nm</td>
</tr>
<tr>
<td>Particle Dispersion</td>
<td>Well-dispersed</td>
</tr>
</tbody>
</table>
percentages of each component are 19.3% for C, 48.1% for O, and 32.6% for Si. The presence of C suggests that the functionalization process was successful and that the 3-methacryloxypropyltrimethoxysilane molecules have been covalently bonded to the surface of the SiO$_2$ nanoparticles. The weight percentage of Si is slightly lower than that of the unfunctionalized SiO$_2$ due to the presence of a carbon skeleton in the structure. Overall, the EDX analysis confirms the successful functionalization of SiO$_2$ nanoparticles with 3-methacryloxypropyltrimethoxysilane (19).

**STEM extract@SiO$_2$ MPS nanoparticles**

**FTIR**

A. FTIR of STEM

The FTIR spectrum of the extracted organic molecules from the plant stem reveals absorption bands at various wavenumbers seen in (Figure S5 and Table S3). 2-Methoxy-4-vinylphenol is identified from bands at 1600 cm$^{-1}$ (C=C stretching) and 3410 cm$^{-1}$ (hydroxyl stretching). Ethyl phthalate is identified from bands at 1639 cm$^{-1}$ and 1620 cm$^{-1}$ (C=O stretching). 1-(N)-hydroxyethyl-9-phenyl-4,5,6,7-tetrahydro[1]benzothieno[2,3-c]pyridine is identified from the band at 1458 cm$^{-1}$ (phenyl group deformation). Decanedioic acid, bis(2-ethylhexyl) ester is identified from the band at 1770 cm$^{-1}$ (C=O stretching). Octasiloxane,1,1,3,3,5,5,7,7,9,9,11,13,13,15,15-hexamethyldecamethyl is identified from bands at 779 cm$^{-1}$ (Si-O-Si rocking) and 470 cm$^{-1}$ (Si-O-Si bending). 2-Undecanone, 6,10-dimethyl is identified from the band at 1072 cm$^{-1}$ (C-C stretching) and 3410 cm$^{-1}$ (exocyclic O-H stretching). The peak at 1099 cm$^{-1}$ is attributed to the Si-O-Si bond's bending vibration in the SiO$_2$-3-methacryloxypropyltrimethoxysilane hybrid material. The 690 cm$^{-1}$ and 651 cm$^{-1}$ peaks are attributed to the Si-O-Si bond's bending vibration in the SiO$_2$-3-methacryloxypropyltrimethoxysilane hybrid material. Finally, the peak at 470 cm$^{-1}$ corresponds to the Si-O-Si stretching vibration in the SiO$_2$-3-methacryloxypropyltrimethoxysilane mixed material (22).

**XRD of STEM extract-SiO$_2$ MPS nanoparticles**

The XRD pattern of SiO$_2$-3-methacryloxypropyltrimethoxysilane-stem (Figure 5) extract shows a diffraction peak at 20 value of 22.5974°, corresponding to a d-spacing of 4.31000Å. The peak's FWHM (full width at half maximum) is 0.900°2θ, and the crystallite size is 9.10 nm (19). Comparing the two XRD patterns of SiO$_2$-3-methacryloxypropyltrimethoxysilane and SiO$_2$-3-methacryloxypropyltrimethoxysilane-stem extract, we can see that both samples have similar crystallite sizes, but there is a slight difference in the position and width of their diffraction peaks. This could be due to the difference in the crystal structure of the two samples, which may be attributed to the stem extract in the SiO$_2$-3-methacryloxypropyltrimethoxysilane-stem.

**SEM of MPS-stem extract nanoparticles**

The SEM analysis of SiO$_2$-3-methacryloxypropyltrimethoxysilane-stem extract (Figure 6 and Table 4) shows an average particle size range of 24.4-37.15 nm, which is...
slightly larger than the particle size range of SiO$_2$-3-methacryloxypropyltrimethoxysilane (17-36.43 nm). Adding the stem extract has contributed to the increase in particle size, possibly due to agglomeration or clustering of the nanoparticles. However, the particles still maintain a spherical shape and are well dispersed, indicating that the stem extract did not significantly affect the overall morphology or dispersion of the nanoparticles. Overall, SEM analysis provides valuable information on nanoparticles' morphology and size distribution, which can help determine their suitability for various applications such as drug delivery, catalysis, and sensing.

The EDX analysis of SiO$_2$-3-methacryloxypropyltrimethoxysilane-stem extract (Figure S7) shows the presence of carbon (C), nitrogen (N), silicon (Si), and oxygen (O) elements. The weight percentages of each component are 19.0% for C, 4.2% for N, 47.8% for O, and 29.0% for Si.

Compared to the EDX analysis of SiO$_2$-3-methacryloxypropyltrimethoxysilane-stem extract, the weight percentage of carbon in SiO$_2$-3-methacryloxypropyltrimethoxysilane-stem extract is slightly lower (19.0% vs. 19.3%), indicating that the functionalization process was successful in both cases the proof of the functionalization was the complete appearance of the organic content in the EDX. The weight percentage of silicon in SiO$_2$-3-methacryloxypropyltrimethoxysilane-stem extract is slightly higher (29.0% vs. 32.6%) compared to SiO$_2$-3-methacryloxypropyltrimethoxysilane, which may be due to the addition of stem extract. The presence of nitrogen in SiO$_2$-3-methacryloxypropyltrimethoxysilane-stem extract indicates the presence of the stem extract, which is not present in SiO$_2$-3-methacryloxypropyltrimethoxysilane-stem extract.

Overall, the EDX analysis confirms the successful functionalization of SiO$_2$ nanoparticles with 3-methacryloxypropyltrimethoxysilane and the successful addition of stem extract to form SiO$_2$-3-methacryloxypropyltrimethoxysilane-stem extract.

**FRUIT extract@SiO$_2$ MPS nanoparticles**

**FTIR**

A. FTIR of Fruit

Based on the FTIR spectrum (Figure S8 and Table S5), the identified functional groups are as follows:

- 5,4'-Dimethoxy-2-methylbibenzyl: C-H stretching (2931 cm$^{-1}$), aromatic ring (1624 cm$^{-1}$), C-H bending (1404 cm$^{-1}$), C-O bond (1076 cm$^{-1}$), 4,7,7-Trimethylbibenzyl (2931 cm$^{-1}$), aromatic ring (1624 cm$^{-1}$), C-H bending (1404 cm$^{-1}$), C-O bond (1076 cm$^{-1}$), 4,7,7-Trimethylbibenzyl (1728 cm$^{-1}$), C-O bond (1076 cm$^{-1}$), Urea (3479 cm$^{-1}$), C=O stretching (1720 cm$^{-1}$), C=C stretching (1624 cm$^{-1}$), C-N stretching (1624 cm$^{-1}$), C-N bending (1404 cm$^{-1}$), 4,7,7-Trimethylbibenzyl (1728 cm$^{-1}$), C-O bond (1076 cm$^{-1}$), Urea (3479 cm$^{-1}$), C=O stretching (1720 cm$^{-1}$), C=C stretching (1624 cm$^{-1}$), C-N stretching (1624 cm$^{-1}$), C-N bending (1404 cm$^{-1}$), 4,7,7-Trimethylbibenzyl (1728 cm$^{-1}$), C-O bond (1076 cm$^{-1}$), Urea (3479 cm$^{-1}$), C=O stretching (1720 cm$^{-1}$), C=C stretching (1624 cm$^{-1}$), C-N stretching (1624 cm$^{-1}$), C-N bending (1404 cm$^{-1}$).

Linoleic acid: C-H stretching (2931 cm$^{-1}$), carbonyl (C=O) stretching (1728 cm$^{-1}$), C=C bond (1624 cm$^{-1}$), CH2 bending (1404 cm$^{-1}$ (23).

B. FTIR of FRUIT extract@SiO$_2$ MPS nanoparticles

The FTIR spectrum of SiO$_2$-3-methacryloxypropyltrimethoxysilane-fruit extract see (Figure S9 and Table S6) reveals critical absorption bands: O-H stretching (3479 cm$^{-1}$), C-O stretching (1720 cm$^{-1}$), C=C stretching (1635 cm$^{-1}$), Si-O-C stretching (1103 cm$^{-1}$), and Si-O-Si bending (466 cm$^{-1}$). These bands confirm the presence of hydroxyl, carbonyl, double bonds, and siloxane bonds, indicating successful synthesis (24).

**XRD of FRUIT extract@SiO$_2$ MPS nanoparticles**

The XRD analysis of SiO$_2$-3-methacryloxypropyltrimethoxysilane-fruit extract in (Figure 7) provided the following information: Peak position: 21.6334°2θ Full width at half maximum (FWHM): 0.9283 Estimated crystallite size: 9.10 nm. Comparison with SiO$_2$-methacryloxypropyltrimethoxysilane revealed a slight shift in peak position and d-spacing for SiO$_2$-methacryloxypropyltrimethoxysilane-fruit extract. However, the FWHM values were comparable, indicating similar crystallinity. The estimated crystallite size was slightly smaller in the fruit extract composite. These differences suggest that the presence of the fruit extract may have influenced the crystallinity and particle size of the composite material.

**SEM and EDX of MPS-fruit extract nanoparticles**

SEM (Scanning Electron Microscopy) analysis of SiO$_2$-methacryloxypropyltrimethoxysilane-fruit extract in (Figure 8 and Table 5) revealed a particle size range of 22.39-34.00 nm. Comparing this with the precursor material SiO$_2$-methacryloxypropyltrimethoxysilane, which exhibited an average particle size range of 17-36.43 nm, we can observe that the particle sizes in the fruit extract are similar. Furthermore, this is larger than the crystallite size estimated from the XRD pattern, which was 9.10 nm. It is not uncommon for there to be differences in particle size of the nanoparticles due to the presence of the fruit extract.
Table 5. SEM data of SiO\textsubscript{2}-3-methacryloxypropyltrimethoxysilane-fruit extract.

<table>
<thead>
<tr>
<th>Characteristic</th>
<th>Observations</th>
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<tr>
<td>Average Particle Size Range</td>
<td>22.39-34.00 nm</td>
</tr>
<tr>
<td>Particle Shape</td>
<td>Irregular spherical shape</td>
</tr>
<tr>
<td>Dispersion</td>
<td>Well dispersed</td>
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</tbody>
</table>

Figure 8. SEM of MPS-fruit extract nanoparticles.

size estimates between XRD and SEM techniques due to differences in the measurement methods and assumptions used. However, the SEM results indicate that the particles in SiO\textsubscript{2}-methacryloxypropyltrimethoxysilane-fruit extract are relatively small and uniform, which is desirable for many applications.

The EDX analysis of SiO\textsubscript{2}-3-methacryloxypropyltrimethoxysilane-fruit extract in (Figure S10) revealed the presence of carbon (C), nitrogen (N), silicon (Si), and oxygen (O) elements. The weight percentages of these elements are as follows: 19.5% for C, 4.4% for N, 48.0% for O, and 28.0% for Si. C and Si indicate the successful functionalization of SiO\textsubscript{2} nanoparticles with 3-methacryloxypropyltrimethoxysilane, as is seen in the supplementary document. The weight percentage of Si is slightly lower than that of unfunctionalized SiO\textsubscript{2}, which is expected. The presence of N suggests the adsorption of nitrogen-containing compounds from the fruit extract onto the nanoparticle surface during synthesis.

**Characterization of ceramic nanoparticles and their nanocomposites**

**Characterization of bioceramic**

The components of bioceramic sealer are ZrO\textsubscript{2} nanoparticles: Zirconia (ZrO\textsubscript{2}) nanoparticles, as mentioned earlier, are ceramic materials known for their biocompatibility and mechanical properties. Zirconia nanoparticles can contribute to the overall strength and stability of the sealer.

**Calcium silicate:** Calcium silicate compounds have bioactive and sealing properties, such as tricalcium silicate (Ca\textsubscript{3}SiO\textsubscript{5}) or dicalcium silicate (Ca\textsubscript{2}SiO\textsubscript{4}). As mentioned, calcium silicate can absorb moisture from the surrounding tissues to form Calcium Aluminate Hydrate (CAH) gel and Calcium Silicate Hydrate (CSH) gel, which aid in sealing the root canal.

**Aluminum silicate:** Aluminum silicate, a compound containing aluminum and silicon, may have specific functions within the formulation of the sealer. Its precise role may vary depending on the manufacturer’s design, and it can contribute to the sealer’s physical or chemical properties.

**Ca(OH)\textsubscript{2}** (Calcium hydroxide): Calcium hydroxide is a well-known dental material with antimicrobial and bioactive properties. Its presence in the sealer formulation suggests it may contribute to the antimicrobial activity and biocompatibility of META biomed Ceraseal (3).

**FTIR**

The FTIR spectrum of the ceramic material in (Figure S11 and Table S7) reveals characteristic bands: O-H stretching (3383 cm\textsuperscript{-1}), C-H stretching (2924 cm\textsuperscript{-1} and 2877 cm\textsuperscript{-1}), C=O stretching (1654 cm\textsuperscript{-1}), aromatic C=C stretching (1577 cm\textsuperscript{-1}), CH\textsubscript{3} deformation (1473 cm\textsuperscript{-1}), C-H bending (1350 cm\textsuperscript{-1}), Si-OH stretching (1103 cm\textsuperscript{-1}), Zr-OH stretching (1064 cm\textsuperscript{-1}), Si-O stretching (945 cm\textsuperscript{-1}), Si-O-Ca or Si-O-Al bending (528 cm\textsuperscript{-1}), and Zr-O bending (420 cm\textsuperscript{-1}). These bands indicate the presence of hydroxyl, organic compounds, silicate, and zirconium-based species in the ceramic material (25).

**XRD**

Based on the given composition of Zirconia (ZrO\textsubscript{2}), calcium silicate, aluminum silicate, and calcium hydroxide, the XRD peaks can be characterized as follows:

1. 28.68°: This peak could be attributed to calcium silicate or Ca(OH), in the mixture.
2. 30.66°, 35.50, 41.71°, 50.72, 60.29°, 63.22°, and 74.34°: This peak may be related to the crystalline phase of Zirconia (ZrO\textsubscript{2}) in the mixture, which corresponds to the phases 101, 002, 102, 200, 211, 202 and 220, respectively (26).
3. 32.02° and 33°: presence of unknown component.
4. 34.84°: This peak corresponds to a crystallographic phase associated with calcium silicate (27). See (Figure 9 and Table 6).

**SEM and EDX**

The SEM characterization of the bioceramic sealer in (Figure 10 and Table 7) shows that the particles have an irregular morphology and are well-dispersed in the sample. The average particle sizes are 60–120 nm, indicating that the sample contains a range of particle sizes. Furthermore, this suggests that the particles are relatively small, and their size distribution may be relatively narrow. It is worth

**Table 6. XRD data of bioceramic.**

<table>
<thead>
<tr>
<th>Pos. [°2Th.]</th>
<th>FWHM [°2Th.]</th>
<th>Particle size [nm]</th>
<th>Average particle size [nm]</th>
</tr>
</thead>
<tbody>
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<td>28.6818</td>
<td>0.2460</td>
<td>34.84</td>
<td></td>
</tr>
<tr>
<td>30.6687</td>
<td>0.2952</td>
<td>29.17</td>
<td></td>
</tr>
<tr>
<td>32.0217</td>
<td>0.1968</td>
<td>43.90</td>
<td></td>
</tr>
<tr>
<td>32.6546</td>
<td>0.2460</td>
<td>35.17</td>
<td></td>
</tr>
<tr>
<td>34.8426</td>
<td>0.1968</td>
<td>44.22</td>
<td></td>
</tr>
<tr>
<td>35.5045</td>
<td>0.3444</td>
<td>25.32</td>
<td></td>
</tr>
<tr>
<td>41.7180</td>
<td>0.7872</td>
<td>11.29</td>
<td></td>
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<tr>
<td>50.7236</td>
<td>0.3444</td>
<td>26.68</td>
<td></td>
</tr>
<tr>
<td>60.2961</td>
<td>0.6888</td>
<td>13.94</td>
<td></td>
</tr>
<tr>
<td>63.2272</td>
<td>0.4920</td>
<td>19.82</td>
<td></td>
</tr>
<tr>
<td>74.3434</td>
<td>1.2000</td>
<td>8.68</td>
<td></td>
</tr>
</tbody>
</table>
noting that these particles may be present due to zirconia.

Based on the given weight percentages in (Figure S12), the composition and positions of the elements in the bioceramic sample can be determined. The bioceramic contains zirconium (40.9%) in the form of zirconium dioxide (ZrO₂), which is known for its high hardness and biocompatibility. Oxygen (25.7%) is present in various compounds, including ZrO₂, calcium hydroxide (Ca(OH)₂), aluminum silicate, and calcium silicate. Calcium (16.6%) is found in Ca(OH)₂ and calcium silicate, both commonly used in bioceramics for bone regeneration. Carbon (8.6%) suggests including an organic component, likely from binders or additives. Silicon (3.2%) is present in aluminum and calcium silicate, critical parts of silicate-based materials. Aluminum (0.7%) is found in aluminum silicate, providing desirable properties in ceramics. The peak positions for each element in X-ray fluorescence spectroscopy indicate typical values, such as Zr (2.04 keV), O (0.53 keV), Ca (3.69 keV), C (0.28 keV), Si (1.74 keV), and Al (1.49 keV). While other elements may be present but not accurately detected, the measurements show that zirconium is the most abundant element, followed by Calcium, silicon, and aluminum. The ingredients are evenly distributed throughout the composition, as shown in the map.

Ceramic-SiO₂-MPS-Fruit extract

FTIR of ceramic-SiO₂-MPS-Fruit extract

The FTIR spectrum of the ceramic-SiO₂-MPS-Fruit extract in (Figure S13 and Table S8) exhibits characteristic bands: O-H stretching (3394 cm⁻¹), C-H stretching (2904 cm⁻¹ and 2873 cm⁻¹), C=O stretching (1716 cm⁻¹), C=C stretching (1577 cm⁻¹ and 1654 cm⁻¹), CH₃ deformation (1477 cm⁻¹), C-H bending (1350 cm⁻¹), Si-OH stretching (1099 cm⁻¹), Zr-OH stretching (1099 cm⁻¹), Si-O stretching (945 cm⁻¹), Si-O-Ca or Si-O-Al bending (590 cm⁻¹), and Zr-O bending (420 cm⁻¹). These bands indicate the presence of hydroxyl, organic compounds, carbonyl groups, aromatic compounds, methyl groups, alkane groups, silanol groups, silicate groups, zirconium hydroxide, and zirconium oxide in the material, reflecting its complex composition resulting from the combination of ceramic and SiO₂-MPS-Fruit extract (24).

XRD of ceramic-SiO₂-MPS-Fruit extract

Based on the given composition of zirconia (ZrO₂), calcium silicate, aluminum silicate, and calcium hydroxide, the XRD peaks can be characterized as follows:

1. 21.35 with FWHM 1.03595: This peak attributed to the amorphous phase of SiO₂-MPS-Fruit extract
2. 28.80°: This peak could be attributed to calcium silicate or Ca(OH)₂ in the mixture.
3. 30.65°, 35.55, 41.83°, 50.69, 60.21°, 63.05° and 74.47°: This peak may be related to the crystalline phase of Zirconia (ZrO₂) in the mixture, which corresponds to the phases 101, 002, 102, 200, 211, 202 and 220, respectively.
4. 31.97°: an unknown component that could be a thickening agent.
5. 34.85°: This peak corresponds to a crystallographic phase associated with calcium silicate. See Figure 11 and Table 8.

XRD of Bioceramic

FTIR of Bioceramic

Table 7. SEM data of bioceramic.

<table>
<thead>
<tr>
<th>Characteristic</th>
<th>Observations</th>
</tr>
</thead>
<tbody>
<tr>
<td>Morphology</td>
<td>irregular</td>
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<tr>
<td>Particle Size Range</td>
<td>60-120 nm</td>
</tr>
<tr>
<td>Particle Dispersion</td>
<td>Well-dispersed</td>
</tr>
</tbody>
</table>

Table 8. XRD data of ceramic-SiO₂-MPS-Fruit extract.

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>21.3500</td>
<td>1.03595</td>
<td>8.16</td>
<td></td>
</tr>
<tr>
<td>28.8034</td>
<td>0.3444</td>
<td>24.89</td>
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<td>30.6513</td>
<td>0.3936</td>
<td>21.87</td>
<td></td>
</tr>
<tr>
<td>31.9758</td>
<td>0.3936</td>
<td>21.95</td>
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<tr>
<td>34.8553</td>
<td>0.3000</td>
<td>29.01</td>
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<td>35.5528</td>
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<td>22.15</td>
<td>19.68</td>
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<tr>
<td>41.8304</td>
<td>0.3936</td>
<td>22.58</td>
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<tr>
<td>50.6994</td>
<td>0.3444</td>
<td>26.68</td>
<td></td>
</tr>
<tr>
<td>60.2138</td>
<td>0.4920</td>
<td>19.51</td>
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<tr>
<td>63.0571</td>
<td>0.7872</td>
<td>12.38</td>
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</tr>
<tr>
<td>74.4752</td>
<td>1.4400</td>
<td>7.24</td>
<td></td>
</tr>
</tbody>
</table>
SEM and EDX of ceramic-SiO$_2$-MPS-Fruit extract

The prepared ceramic-SiO$_2$-MPS-fruit extract composite material exhibited regular nanosphere morphology. This indicates that the particles in the composite possessed a more spherical and uniform shape than the irregular ceramic particles. Additionally, the particle size range of the composite material was reduced to 23-46 nm, indicating a decrease in particle size when compared to the original ceramic particles. This size reduction could be attributed to including SiO$_2$, MPS, and fruit extract in the composite. Moreover, the particle dispersion in the composite material was described as well-dispersed, suggesting an excellent distribution of particles throughout the composite without any significant clustering or agglomeration. See (Figure 12 and Table 9).

The EDX analysis of the ceramic-SiO$_2$-MPS-Fruit extract composite (Figure S14) provided the following elemental composition: Based on the EDX results, it can be observed that Zr (Zirconium) is present in the composite, accounting for 17.67% of the total weight. This suggests that Zr is a constituent element of the composite material. Furthermore, the EDX analysis implies that Zr is predominantly located on the surface of the composite. The decrease in Zr weight percentage in the composite compared to the bioceramic (40.9) is likely influenced by the addition of SiO$_2$-MPS-Fruit extract. The SiO$_2$-MPS and Fruit extract components may have displaced or substituted some of the Zr atoms on the surface, resulting in a lower weight percentage of Zr. This can occur due to the different chemical interactions and bonding characteristics between Zr and the other components of the composite.

Furthermore, the increase in carbon content observed in the ceramic-SiO$_2$-MPS-Fruit extract composite compared to the original ceramic material is likely attributed to adding the Fruit extract component.

Ceramic-SiO$_2$-MPS-Stem extract

FTIR of ceramic-SiO$_2$-MPS-Stem extract

The FTIR spectrum of the ceramic-SiO$_2$-MPS-Stem extract in (Figure S15 and Table S8) reveals characteristic bands indicating the presence of various functional groups and compounds. These include hydroxyl groups (3371 cm$^{-1}$), C-H and C=O groups in organic compounds (2924 cm$^{-1}$, 2877 cm$^{-1}$, 1716 cm$^{-1}$), vinyl and aromatic groups (1639 cm$^{-1}$, 1581 cm$^{-1}$), alkane groups (1469 cm$^{-1}$, 1354 cm$^{-1}$), silicate groups (1296 cm$^{-1}$, 941 cm$^{-1}$), silanol and zirconium hydroxide groups (1103 cm$^{-1}$, 1064 cm$^{-1}$), zirconium oxide (420 cm$^{-1}$), and specific bonds in the hybrid materials (Si-O-Ca/Al, Si-O-Si). The analysis suggests a complex composition resulting from the combination of ceramic, SiO$_2$-MPS-Stem extract, and specific organic molecules used in the Preparation (21).

XRD of ceramic-SiO$_2$-MPS-Stem extract

Based on the given composition of zirconia (ZrO$_2$), calcium silicate, aluminum silicate, and calcium hydroxide, the XRD peaks can be characterized as follows:

1. 20.77° with FWHM 0.919: This peak is attributed to the amorphous phase of SiO$_2$-MPS-Stem extract.
2. 28.69°: This peak could be attributed to calcium silicate or Ca(OH)$_2$ in the mixture.
3. 30.54°, 35.47°, 41.67°, 50.17°, 60.15°, 63.11°, and 74.74°: This peak may be related to the crystalline phase of Zirconia (ZrO$_2$) in the mixture, which corresponds to the phases 101, 002, 102, 200, 211, 202 and 220, respectively.
4. 31.87° and 33°: an unknown component that could be
a thickening agent.

34.67°: This peak corresponds to a crystallographic phase associated with calcium silicate. See (Figure 13 and Table 10).

**SEM and EDX of ceramic-SiO₂-MPS-Stem extract**

The SEM analysis of the ceramic material revealed an irregular morphology with particles ranging in size between 60 and 120 nm. These ceramic particles were well-dispersed without significant agglomeration because of the shape of the nanoparticles. However, after incorporating SiO₂-MPS-Stem extract into the composite material, the particle morphology changed to quasi-regular nanospheres. This indicated a more uniform and spherical shape than the irregular ceramic particles. See (Figure 14 and Table 11).

Based on the EDX results, it can be observed that Zr (Zirconium) is present in the composite with 19.2% of the total weight. This suggests that Zr is a constituent element of the composite material. Furthermore, the EDX analysis implies that Zr is predominantly located as the core of the composite, not as in the case of the composite containing fruit extract. Regarding the decrease in Zr weight percentage in the composite compared to the ceramic (40.9), it is likely influenced by the addition of SiO₂-MPS-Stem extract. In summary, the EDX analysis confirmed the presence of Zr in the ceramic-SiO₂-MPS-Stem extract composite, with a higher concentration on the surface. The decrease in Zr weight percentage may be attributed to incorporating SiO₂-MPS-Stem extract, which can influence the distribution and interaction of Zr atoms in the composite (See Figure S16).

**Discussion**

Nanotechnology, manipulating matter at the nanoscale, has emerged as a promising field with diverse applications in various sectors, including dentistry. The integration of nanotechnology into dental practices has the potential to revolutionize diagnosis, treatment, and preventive care (14). This discussion essay explores the applications of nanotechnology in dentistry, highlighting its impact on oral health and patient care.

In the current study, Fourier transforms infrared (FTIR) spectroscopy was used, a powerful analytical technique widely used to identify and quantify chemical compounds in a sample. It works by measuring the absorption or transmission of infrared radiation by the model, which is then used to generate a spectrum that provides information about the chemical bonds in the material; based on the results of the present study, the functional chemical bonds produced by the core-shell (fruit-silica and stem-silica) were similar and showed the presence of the same band peaks in the final product which is an indication for the perfect chemical reaction of the organic components of the Berberis vulgaris extracts and the bioceramic sealer which is similar to the results of a study made by Kazemi poor in Iran (28).

X-ray diffraction (XRD) is commonly used to analyze materials’ crystal structure and crystallinity. In the case of silica nanoparticles, XRD can determine if the nanoparticles are crystalline or amorphous and estimate the size of any crystalline regions present (29). The Scherrer equation can estimate the crystallite size, relating FWHM to crystallite size. Assuming a spherical shape, this suggests that the silica nanoparticles are relatively amorphous with some crystallinity. The crystallite sizes for the [101] and [200] peaks are estimated to be 10.6 nm and 10.8 nm, respectively. These values indicate that the nanoparticles have a small crystallite size, consistent with typical silica nanoparticles, which is a critical point to be considered as the size of the nanoparticle is as tiny as possible, as indicated by Nikmah et al. (16), according to the results of the present study the XRD results showed that both SiO₂-MPS-Stem extract and SiO₂-MPS-fruit extract have amorphous and crystallinity phase.

Several methodologies have been introduced to evaluate the novel core products’ structure and size; two of the most common methods are Scanning Electron Microscopy(SEM) and Energy-Dispersive X-ray Spectroscopy (EDX), the SEM of silica nanoparticles showed the spherical shape of the nanoparticles, which is advantageous for many applications as it provides a large surface area to volume ratio, which can enhance reactivity and facilitate surface modification. The well-dispersed nature of the nanoparticles is also essential, as it suggests that

---

**Table 10. XRD data of ceramic-SiO₂-MPS-Stem extract.**

<table>
<thead>
<tr>
<th>Pos. [°2Th.]</th>
<th>FWHM [°2Th.]</th>
<th>Particle size [nm]</th>
<th>Average particle size [nm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>20.77</td>
<td>0.919</td>
<td>9.19</td>
<td></td>
</tr>
<tr>
<td>28.6913</td>
<td>0.2952</td>
<td>29.03</td>
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<td>30.5411</td>
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<tr>
<td>31.8712</td>
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<td>34.6744</td>
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</tbody>
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**Table 11. SEM data of ceramic-SiO₂-MPS-Stem extract.**

<table>
<thead>
<tr>
<th>Characteristic</th>
<th>Observations</th>
</tr>
</thead>
<tbody>
<tr>
<td>Particle Size Range</td>
<td>quasi-regular nanospheres</td>
</tr>
<tr>
<td>Particle Dispersion</td>
<td>40-89 nm</td>
</tr>
<tr>
<td>Characteristic</td>
<td>Observations</td>
</tr>
<tr>
<td></td>
<td>well-dispersed</td>
</tr>
</tbody>
</table>

**Figure 14.** SEM of ceramic-SiO₂-MPS-Stem extract.
the nanoparticles have good stability in solution and can be easily incorporated into various materials or systems. According to the results of the present study, the ESM and EDX SiO$_2$-MPS-fruit extract showed that the Preparation of the ceramic-SiO$_2$-MPS-fruit extract composite resulted in a transformation of the irregular ceramic particles into regular nanospheres with a smaller particle size range (30). The composite material demonstrated a highly uniform and very well-dispersed distribution of particles; EDX analysis confirmed the presence of Zr in the ceramic-SiO$_2$-MPS-Fruit extract composite, with a higher concentration on the surface. The decrease in Zr weight percentage may be attributed to incorporating SiO$_2$-MPS-Fruit extract, which can influence the distribution and interaction of Zr atoms in the composite. It is recommended to test the extracts of other plants as well as different plant parts for the synthesis of nanoparticles (31-34).

ESM and EDX analysis of SiO$_2$-MPS-Stem extract showed that the particle size range of the composite material was reduced to 40-89 nm, suggesting a decrease in particle size compared to the original ceramic particles. This size reduction could be attributed to the additional components present in the composite. Despite the morphological and size changes, the composite material maintained good dispersion, indicating an even distribution of particles without significant agglomeration. In summary, the Preparation of the ceramic-SiO$_2$-MPS-Stem extract composite resulted in the transformation of irregular ceramic particles into quasi-regular nanospheres with a smaller size while preserving good dispersion characteristics.

This novel approach investigated synthesizing a new core-shell structure from nanoparticles enhanced by herbal products from Berberis vulgaris to produce and characterize a novel product that can be used in different medical and dental fields.

In this study, the focus was on improving the mechanical and biological properties of bioceramic mixtures. The active compounds extracted from Berberis vulgaris stems and fruits were analyzed using GC-MS and FTIR, providing insights into their composition. These extracts were then used to synthesize novel nanocomposites, namely SiO$_2$-MPS-stem extract and SiO$_2$-MPS-fruit extract. Characterization techniques such as SEM, EDX mapping, FTIR, and XRD were employed to analyze these composites, confirming the successful coating of silica with the extracts and the formation of core-shell nanostructures with particle sizes below 60 nm. The composites were incorporated into bioceramics for dental root fillings, maintaining an equal weight ratio. The characterized ceramic overlays exhibited nanoscale size, not exceeding 70 nanometers. Notably, the results indicated a core-shell configuration for the nanomaterials, with the shell component consisting of the bioceramic in the case of bioceramic-SiO$_2$-MPS-fruit extract. At the same time, the reverse was observed for bioceramic-SiO$_2$-MPS-stem extract. These findings demonstrate the potential of utilizing bioceramic mixtures enhanced with Berberis vulgaris extracts in dental applications, as they can achieve improved mechanical and biological properties by creating well-defined nanocomposites. Further research and testing are needed to assess their efficacy and safety in dental root-filling procedures.

Consent for publications
The author read and proved the final manuscript for publication.

Availability of data and material
All data generated during this study are included in this published article.

Authors’ contribution
All authors had equal roles in study design, work, statistical analysis, and manuscript writing.

Funding
This article was self-funded by the authors without any institutional or college funding.

Ethics approval and consent to participate
No humans or animals were used in the present research. The Ethics Committee of Hawler Medical University approved the study protocol.

References
12. Piro NS, Hamad SM, Mohammed AS, Barzinjy AA. Green Synthesis Magnete (Fe$_3$O$_4$) Nanoparticles From Rhus coriaria Extract: 274


