

RESEARCH ARTICLE

Novel CaO/polylactic acid nanoscaffold as dental resin nanocomposites and the investigation of physicochemical properties

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Abstract

In this study, for the first time, calcium oxide (CaO)/polylactic acid nanoscaffolds were synthesized by co-precipitation assistant reverse micelles method. The physical and chemical (physicochemical) properties of the structures as dental resin composites were also studied. Nanocomposite materials as primary and basic dental compounds can be conveniently applied as dental filling materials with a high esthetic quality. In this research nanoscaffolds act as a bed for nanoparticles and improve the mechanical and chemical (mechanochemical) properties, CaO nanoparticles were loading in polylactic acid nanoscaffold as a bioactivity polymer for usage in the dental resin composites. Mechanical properties of the dental resin composite containing CaO/polylactic acid nanoscaffold were calculated: the flexural strength (137.2 MPa), modulus (12.9 GPa) and compressive strength (344.2 MPa). Potential of the basic nanoparticle and the products were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), thermogravimetric analysis (TGA), dynamic light scattering (DLS), ultraviolet-visible spectroscopy (UV-visible) and atomic force microscopy (AFM) showed the size of the optimized nanostructures was about 85 to 120 nm. According to TGA results of polylactic acid nanofibers with thermal stability below 300°C these high thermal stability materials can be used as dental resin composites.

KEYWORDS

CaO/polylactic acid, dental nanocomposites, nanoscaffold, reverse micelle

1 | INTRODUCTION

Recently nanodentistry as a new field has had a growing interest in the dental applications of nanotechnology.^[1,2] Nanotechnology improves the efficiency of structures and has recently attracted the attention of many researchers in the sciences due to the unique properties of materials in nanotechnology.^[3,4] Increasing the surface-to-volume ratio of nanoparticles is one of the most important characteristics, which improves the strength and mechanical properties.^[5,6] The most important application of nanoparticles and

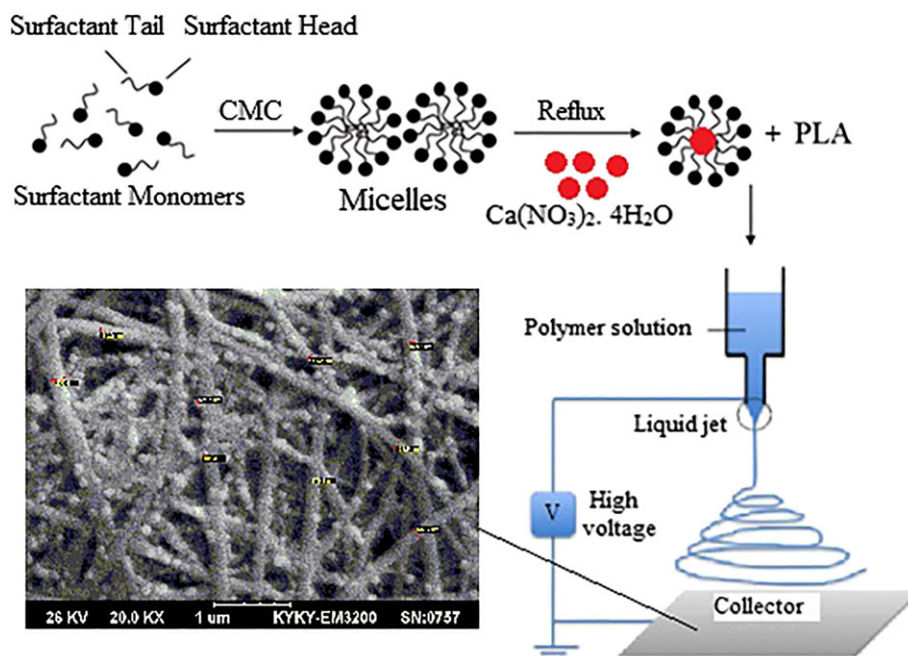
chalcogenide nanocrystals have been in dental composites where the mechanochemical and physicochemical properties can be enhanced by creating a mixture of nanoparticles and a substrate phase as a fixative.^[7-9] Nanotechnology can be utilized in various dental fields by way of antimicrobial agents,^[10] dental instruments,^[11,12] dental material,^[13] clinical dentistry,^[14] and dental resin composites.^[15] In the last few years, nanotechnology has played an important role in improving the clinical performance of dental resin composites.^[16,17] The use of nanocomposite resins improves the bond between the tooth structure and the restorative materials,

which leads to a more stable and natural interface.^[18,19] Previous studies indicate that the use of many kinds of materials for composite resins such as acrylic resins, silicate cements, matrix organic phases as dimethacrylate,^[20] urethane dimethacrylate (UDMA),^[21] triethyleneglycol dimethacrylate (TEGDMA),^[22] bisphenol-A-dimethacrylate (Bis-EMA),^[23] decanediol dimethacrylate (D₃MA)^[24] raises concerns about the mechanical and fracture properties owing to the large size of the particles. Recently, micelles have been shown significant interest and attention as delivery systems for poorly water-soluble drugs. Micelles can be used to deliver hydrophilic and hydrophobic drugs. The micellation process as the *in situ* method is where amphiphilic molecules with hydrophilic/polar head and hydrophobic/non-polar tail form self-assembly structures, many of the common chemical approaches for micelle formation are oil in water emulsion,^[25] solvent evaporation,^[26] solid dispersion^[27] and dialysis methods.^[28] Micelles have high ability to store and protect drug molecules within itself and delivery them according to sensitivity to pH, temperature and concentration of surfactants.^[29] Scaffolds as a platform used to elevate and support structures and materials during the construction, form chemical bonds that improve

mechanochemical and shape stability for many of the compounds. Many researchers have studied various methods and materials as dental resin composites, the summary of this research is indicated in Table 1. In this study we present degradable polymers with nanoscaffold morphological properties which have high mechanical properties compared to nanoparticles in other studies. Resin composites in dentistry are generally composed of three chemical components including an organic matrix part, filler or disperse phase and an organosilane that is a coupling agent for the bond between the filler particles and the organic matrix. The significance of this article is that a new and novel synthesis and preparation of calcium oxide (CaO)/polylactic acid nanoscaffold as a new dental resin composite is presented. In this approach, CaO nanoparticles were prepared with size under 100 nm. Then they were placed in polylactic acid based as a biodegradable and biocomposite polymer. Next, physicochemical properties of the product were studied by X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), dynamic light scattering (DLS), Fourier transform infrared (FTIR), Brunauer–Emmett–Teller (BET) analysis. Scheme 1 indicates the path for the formation of the CaO/polylactic acid nanoscaffold.

TABLE 1 Some useful and extensive research on nanoparticles and nanocomposites as dental resin composites in recent years

Types of nanocomposites	Purpose of use	Synthesis method	Size of nano-emulsion	References
Ceramics		Homogenization	100–130 nm	[31]
Silane	Sorption characteristics	Aerosil	150–180 nm	[32]
Methacryloxypropyltrimethoxysilane	Experimental resin composite	Physical–mechanical	360 nm	[33]
Hydroxyl apatite/ZnO-MgO	Biological properties	Ultrasonic method	400 nm	[34]
Hydroxyapatite, alumina/zirconia	Mechanical properties	Co-precipitation	90 nm	[35]
Curcumin-loaded chitosan	Bacterial properties	Ultrasonic method	35.9 nm	[36]



SCHEME 1 Summary of experimental route for the preparation of nanoscaffold as dental resin composites

2 | EXPERIMENTAL

2.1 | Materials and characterization

All chemicals were of the highest purity and were not purified any further. The water used in this study was deionized. Calcium nitrate ($\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$) and sodium hydroxide (NaOH) of analytical grade were purchased from Merck Co. (Kenilworth, NJ, USA) and used without any further purification. The non-ionic surfactants used as micelle-forming materials, sorbitan monopalmitate (Span 40), sorbitan monostearate (Span 60), sorbitan oleate (Span 80), were purchased from Fluka (Buchs, Switzerland). The dental restorative resin composite was Heliomolar Flow (Ivoclar Vivodent AG, FL-9494, Schaan, Liechtenstein) that was provided by Sigma-Aldrich (St Louis, MO, USA). The CaO /polylactic acid nanoscaffold was characterized by XRD with the specifications Rigaku D-max C III X-ray diffractometer using Ni-filtered $\text{Cu K}\alpha$ radiation. Distribution size, morphology and nanoscale pictures of products were visualized by SEM (LEO 1455VP), TEM images were obtained on a Philips EM208S transmission electron microscope with an accelerating voltage of 100 kV. Thermal stability of the nanostructure formulations was examined by thermogravimetric analysis (TGA) curves with specifications from room temperature to 800°C with a heating rate of $10^\circ\text{C}/\text{min}$ under an argon atmosphere using a SCINCO thermal gravimeter S-100 (Seoul, Korea). The DLS experiments were performed using the Brookhaven Instruments 90 Plus particle size analyzer in Razi Metallurgical Research Center Iran. To study the surface distribution and porosity and the average heights of the nanoparticles atomic force microscopy (AFM) measurements were done. AFM images were obtained in an electronic nano laboratory at the Technical University of Tehran-Iran (Digital Instrument DualScope™ DS95-50, DME, Denmark). Energy-dispersive X-ray spectroscopy (EDX, 7533, Oxford Instruments, Oxford, UK) was used to calculate the chemical elemental analysis of the prepared nanostructures.

2.2 | Preparation of CaO nanoparticles

At first, we dissolved 0.80 g of $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ in a mixture of deionized water-ethanol (molecular ratio 2:1) and then the pH value was set between 7.5 and 8.3. Next the solution was vigorously stirred with a magnetic stirrer on a heater 55 degree of centigrade for 120 min. After the experimental setup for the synthesis process, the mixture was moved to the reflux system. In another beaker different amounts of concentration of Span 40, Span 60 and Span 80 as surfactant together with cetyltrimethylammonium bromide and sodium dodecyl sulfate as co-surfactant were added dropwise into a 10 mL water deionized solution with stoichiometric ratio of surfactant-co-surfactant of 1:2. By adding the surfactant concentration a critical point (critical micelle concentration, CMC) was achieved. This solution was heated at 50°C for 1 h then added dropwise to the reflux system. After 120 min, a white precipitate was collected by filtration and then the precipitate was washed with absolute ethanol and distilled water several times. The experimental conditions for the prepared CaO nanoparticles are shown in Table 2. Finally, precipitates were dried at room temperature for 4 days.

TABLE 2 Summary of conditions for the synthesis of calcium oxide (CaO)/polylactic acid nanoscaffold composites

Sample number	Surfactant	Co-surfactant	Molar ratio	Temperature ($^\circ\text{C}$)	pH	Size (nm)
1	Span 40	CTAB	1:2	50	6.5	115
2	Span 60	CTAB	1:2	50	6.5	109
3	Span 80	CTAB	1:2	50	6.5	85
4	Span 40	SDS	1:2	50	6.5	119
5	Span 60	SDS	1:2	50	6.5	111
6	Span 80	SDS	1:2	50	6.5	91

Note: CTAB, cetyltrimethylammonium bromide; SDS, sodium dodecyl sulfate.

2.3 | Preparation of CaO /polylactic acid nanoscaffold

In this study, the electrospinning technique was used for the synthesis and preparation of nanoscaffolds. Initially, 20 wt% of polylactic acid in chloroform was prepared by dissolving a weighed amount of polylactic acid in chloroform with constant stirring for 60 min at 50°C . Then, 5 wt% of CaO nanoparticle solution in a ratio 2:1 of chloroform-deionized water was prepared and mixed into the solution of polylactic acid.

Next, an electrospinning device was set up with a high-voltage power supply capable of generating voltages up to 30 kV. The polymer solution was supplied through a 5 mL plastic syringe attached to a tip with an inner diameter of 0.4 mm and the feed rate of the solution was controlled at 1.0 mL/h. The pieces of aluminum sheets were on a rotating metal drum (diameter of 6 cm) as the fiber collector. The distance between the tip of the nozzle and the outer surface of the drum and the rotational speed of the drum setup were 10 cm and 100 rpm, respectively. All of the electrospinning procedures were carried out at 25°C and 120 min. In the final step the system was allowed to cool to room temperature naturally, and the obtained precipitations were collected.

2.4 | Preparation of resin nanocomposites

In this study after the CaO /polylactic acid nanoscaffold was synthesized it was added to Heliomolar Flow (Ivoclar Vivodent AG, FL-9494) resin composites. In order to investigate the mechanical properties of the nanostructures, different concentrations of CaO /polylactic acid nanoscaffold from 0 ppm up to 100 ppm wt% were added to the resin composite. The obtained products were placed in the oven under vacuum at room temperature for 24 h, then put in a refrigerator (4°C).

3 | RESULTS AND DISCUSSION

3.1 | Structural analysis (XRD)

In this study CaO /polylactic acid nanoscaffold as dental resin composite was synthesized through the micellation method assisted by the

electrospinning method. Phase identification of crystals on unit cell dimensions was measured with XRD analysis. XRD pattern of CaO nanoparticles and CaO/polylactic acid nanoscaffold composite are indicating in Figure 1(a,b) respectively. The XRD scanned in the range $10^\circ < 2\theta < 80^\circ$. The results show that nanoscaffold composite crystals included both CaO nanoparticles and polylactic acid nanoscaffold. The XRD analysis indexed as 13.1° , 34.3° , 38.1° , 41.8° , 51.3° , 59.2° and 62.2° could be assigned to the (111), (211), (201), (211), (311), (222), (301) planes. From the full width of the half maximum (FWHM) with the Debay-Scherer equation particle size of CaO/polylactic acid nanoscaffold composite obtained was about 95 nm (equation 1).

$$D_c = K\lambda/\beta \cos\theta \quad (1)$$

where λ is the copper wavelength (1.54056 Å), θ is the Bragg diffraction angle, and β is the half-width of the most intense diffraction peak.

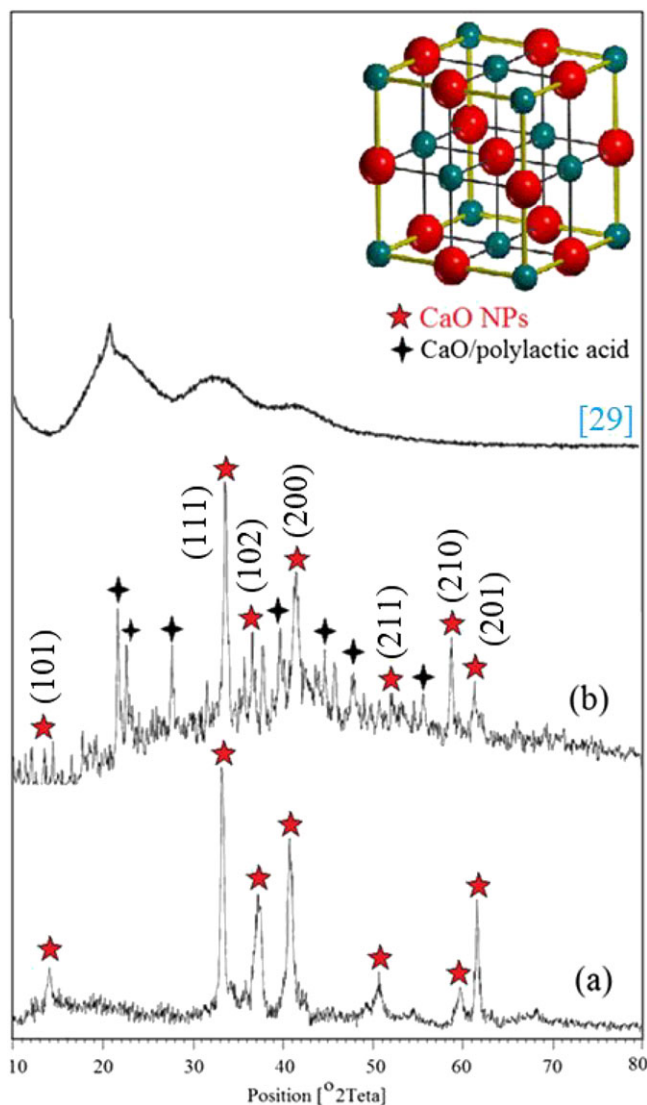


FIGURE 1 X-ray diffraction (XRD) pattern of calcium oxide (CaO) nanoparticles (NPs) (a), CaO/polylactic acid nanoscaffold composite (b) and polylactic acid^[30]

3.2 | Size and surface morphology

To obtain size and morphology data SEM images were used to investigate morphology and particle size distribution. SEM images of polylactic acid nanoscaffold composites, CaO/polylactic acid nanoscaffold composites and DLS diagram of nanoscaffolds at room temperature respectively are displayed in Figure 2(a–c). Polylactic acid nanoscaffold composites show separated structures with suitable distance. Connectivity and continuity of nanofibers are one of the most important features in dental resin composites. It is obvious that CaO nanoparticles in emulsions loaded on polylactic acid nanoscaffold were distributed as a uniform particle size. DLS as a physical method was used to determine the distribution of nanoparticles in solutions and suspensions. This non-destructive and rapid method is used to determine the particle size in the range of a few nanometers to microns.

The DLS diagram of CaO/polylactic acid nanoscaffold show particle size of products about 95–115 nm. TEM image of CaO/polylactic acid nanoscaffold composite with a magnification of 40000 times is shown in Figure 2(d). It is seen that the morphological structure is almost consistent with that observed from SEM images of CaO/polylactic acid nanoscaffold composite. The nanoparticle stabilized in emulsion droplets are small enough to load on polylactic acid nanoscaffold uniformly. Both analyses by SEM and TEM support that the CaO nanoparticles in emulsion are adsorbed on the surface of the polylactic acid nanoscaffold.

AFM has been used to measure the surface properties and mechanical properties such as elastic modulus, which is a measure of the stiffness of a dental resin composite or its tendency to deform elastically under an applied force, or adhesion. AFM images of CaO nanoparticles and CaO/polylactic acid nanoscaffold composites are shown in Figure 3(a,b). The surface properties of the CaO/polylactic acid nanoscaffold were estimated quantitatively by the root mean-squared roughness (R_{rms}) which was obtained from the AFM data image and determined according to the equation (equation 2):

$$R_{rms} = \sqrt{\frac{\sum_{n=1}^N (z_n - \bar{z})^2}{N - 1}} \quad (2)$$

where z_n represents the height of the n th data, $\bar{z}$ is equal to the mean height of z_n in AFM topography, and N is the number of data. For an area of $73.98 \mu\text{m}^2$ the roughness average is 1.94 nm.

According to AFM images, CaO nanoparticles are completely independent which confirms SEM and TEM images. Also, CaO/polylactic acid nanoscaffold composites are obtained as regular and separate from each other fibers. These nanofibers are clearly visible in AFM image with approximate diameter of 95–120 nm. EDX diagram shows the purity and chemical composition of CaO/polylactic acid nanocomposites in Figure 4(c). The presence of C, O and Ca elements confirms that CaO/polylactic nanofibrous composites were synthesized without any impurities or trace elements.

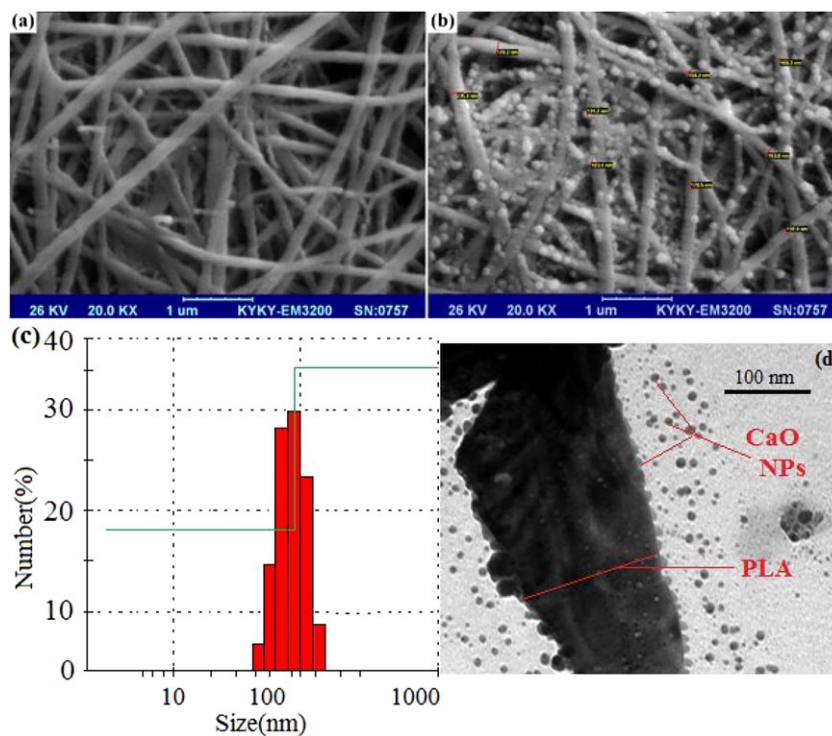


FIGURE 2 Scanning electron microscopy (SEM) images of poly(lactic acid) nanofibers (a), calcium oxide (CaO)/poly(lactic acid) nanoscaffold composite (b), dynamic light scattering (DLS) plot of CaO/poly(lactic acid) nanoscaffold composite (c) and transmission electron microscopy (TEM) image of CaO/poly(lactic acid) nanoscaffold composite (d)

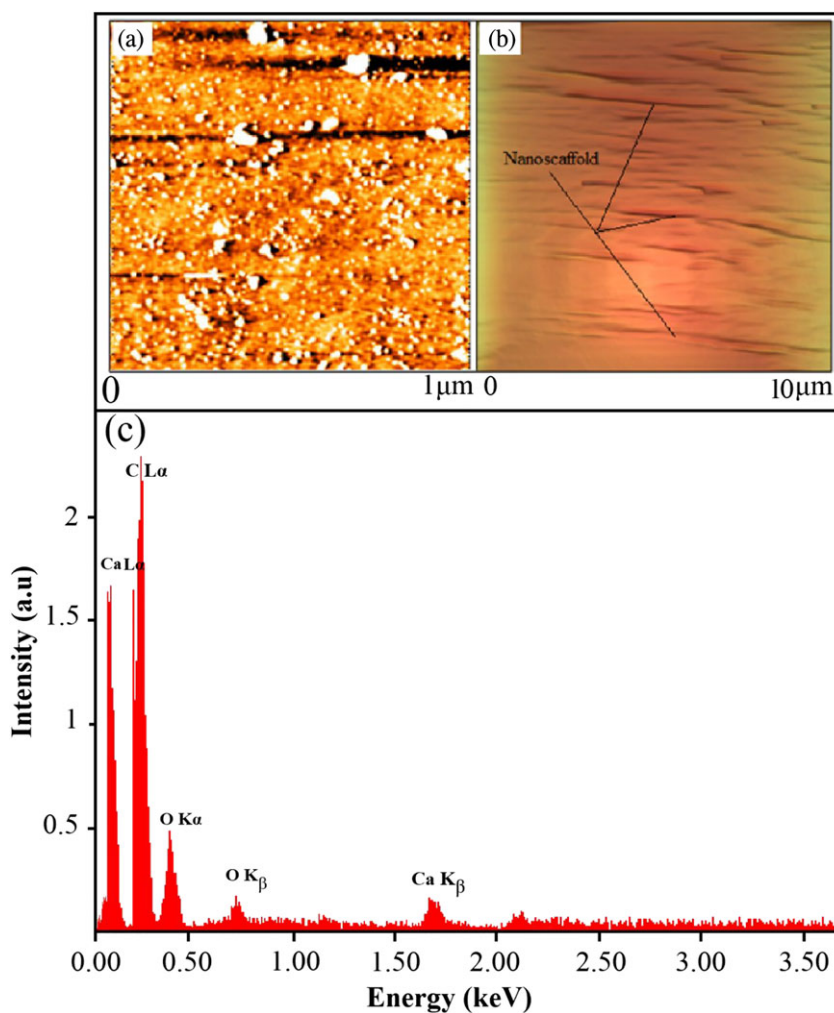


FIGURE 3 Atomic force microscopy (AFM) images of the synthesized calcium oxide (CaO) nanoparticles (NPs) (a), CaO/poly(lactic acid) nanoscaffold composite (b) and energy-dispersive X-ray spectroscopy (EDX) spectrum of the CaO/poly(lactic acid) nanoscaffold (c)

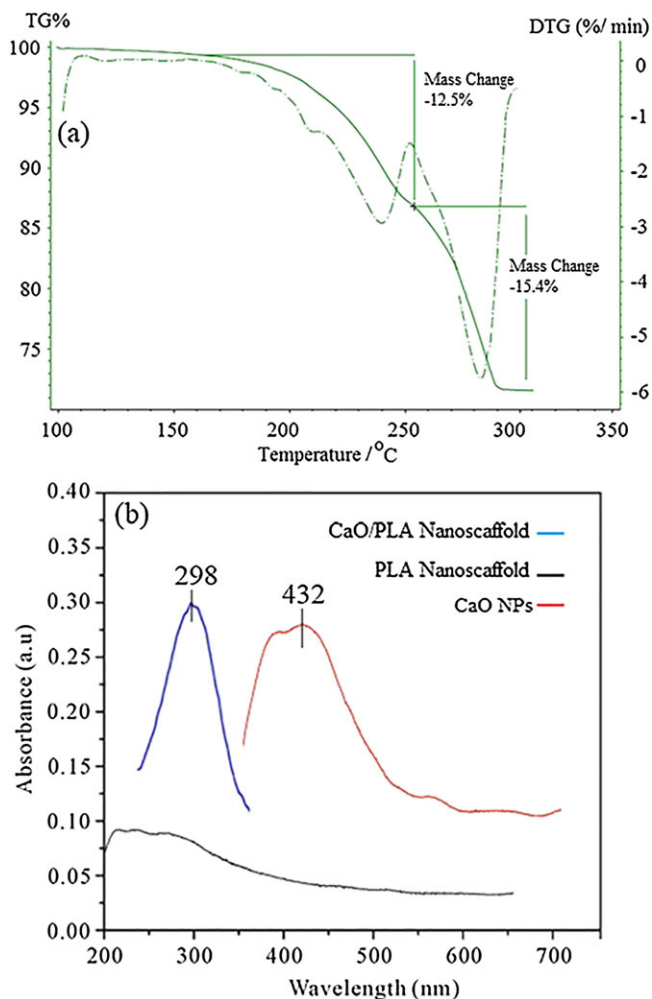


FIGURE 4 Thermogravimetric analysis of the synthesized calcium oxide (CaO)/polylactic acid nanoscaffold composite (a) and the UV-visible spectrum of CaO nanoparticles (NPs) (red line) and CaO/polylactic acid nanoscaffold composite (blue line) and polylactic acid nanofibers (black line) respectively (b)

3.3 | Thermogravimetric analysis (TGA)

The mass of CaO/polylactic acid nanoscaffold was measured over time as the temperature changes with thermal gravimetric analysis (TGA) as a method of thermal analysis in certain temperature is shown in Figure 4(a). The TGA and differential thermogravimetry (DTG) analysis were carried out with a Diamond Perkin Elmer under a nitrogen atmosphere at a heating rate of 10°C/min. Results indicate a small weight loss of about 12.5% at the temperatures around 170 and 250°C which is ascribed to the degradation of CaO nanoparticles in the surfactant phase in micro- and nano-emulsions. Between 250 and 300°C the polylactic acid nanoscaffold decomposes completely with a rapid weight loss of over 15.4%. Therefore, according to TGA results polylactic acid nanofibers can be used for dental resin composites as high thermal stability materials.

3.4 | UV-visible spectrum

The improvement in optical properties without sacrificing mechanical strength and wear resistance is one of the reasons for using nanoparticles as dental resin composites. To evaluate optical activity of products UV-visible spectroscopy is used to measure the attenuation of a beam of light after it passes through a sample or after reflection from a sample surface of the CaO nanoparticles, polylactic acid nanofibers and CaO/polylactic acid nanoscaffold composites as shown in Figure 4(b). By using UV-visible spectroscopy, bandgap can be characterized along with the relative energetic position of sub-bandgap defect states. The optical band gap can be calculated by plotting a graph of $(ah\nu)^2$ as a function of $h\nu$ according to the equation (Equation 3):

$$(ah\nu)^2 = B(h\nu - E_g)^n \quad (3)$$

where B is a constant, ' E_g ' is an optical band gap of CaO and exponent n is the nature of the transition. The UV-visible spectrum for CaO nanoparticles excited at 420 nm can be related to a singly charged oxygen position from the recombination of a photo generated hole with a charge state of the specific defect such as oxygen vacancies in CaO NPs. The blueshift observed in the UV-visible spectrum could be due to diminishing defects in CaO/polylactic acid nanoscaffold composites because of the presence of polylactic acid nanoscaffolds.

3.5 | Mechanical properties

Flexural strength, modulus and compressive strength are the most important mechanical tests for determination of mechanical properties in dental resins. Mechanical properties of the resin composites such as the flexural strength, modulus and compressive strength tests for nanoscaffold composites were measured according to ISO 4049 and ISO 9917 in terms of MPa. All three-point bendings were performed on a specified template with 2 mm × 2 mm × 25 mm dimensions using a universal testing machine (Z20, Zwick Roell, Ulm, Radeberg, Germany) at a cross-head speed of 0.5 mm min⁻¹ as shown in Figure 5. Results indicate that the mechanical properties of the dental resin composite were improved by the addition of CaO/polylactic acid nanoscaffold composites. In this study different concentrations of CaO/polylactic acid nanoscaffold from 0 ppm up to 100 ppm wt% were added to the resin composite.

In the sample of the CaO/polylactic acid nanoscaffold loaded in resin composite with 60 ppm (wt) concentration, the flexural strength (137.2 MPa), modulus (12.9 GPa) and compressive strength (344.2 MPa) were calculated. With the increase of nanoscaffold concentration from the blank sample to 100 ppm (wt) the flexural strength, modulus and compressive strength gradually increased. After 60 ppm (wt) concentrate of CaO/polylactic acid nanoscaffold the mechanical properties of the dental resin composite decreased. The results showed that increasing the concentration of nanoscaffolds causes aggregation in structures. This decrease in property is owing to the dispersion size of the nanostructures in the resin composite.

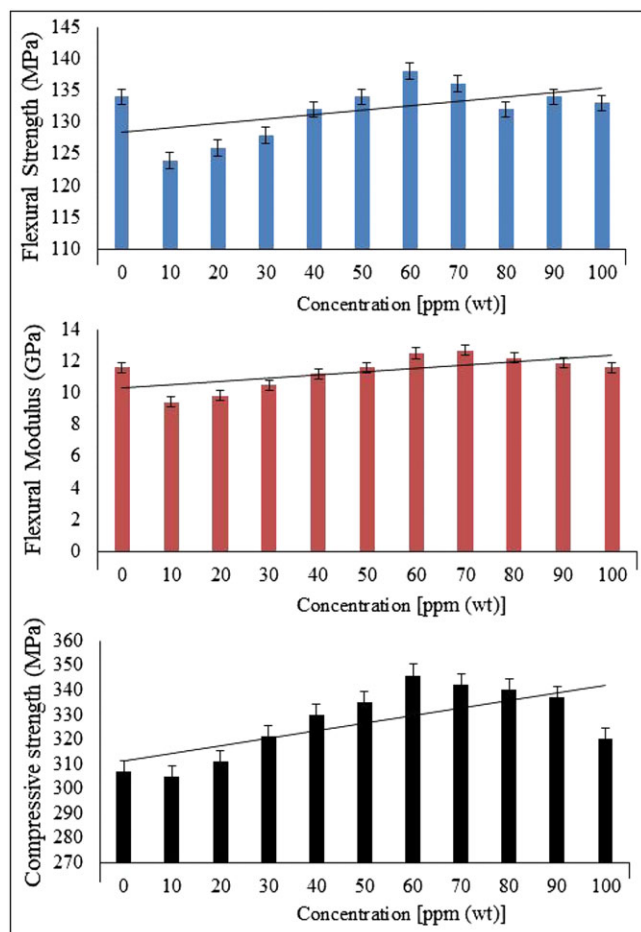


FIGURE 5 Mechanical properties of the final resin composites with different amounts of calcium oxide (CaO)/polylactic acid nanoscaffold. (a) Flexural strength (MPa); (b) flexural modulus (GPa); (c) compressive strength (MPa)

4 | CONCLUSION

Resin-based nanocomposites with improved physical properties show a lot of potential for widespread use in the restoration of cavities in posterior teeth. In this study for the first time, CaO/polylactic acid nanoscaffold composites were synthesized by the co-precipitation assisted microwave method. Polylactic acid was used as a biocompatible and biodegradable polymer for loading CaO nanoparticles. Products were characterized by AFM, DLS, XRD, SEM, TEM, TGA and UV-visible spectrum. The suitable flexural strength, modulus and compressive strength of the nanoscaffold nanocomposites with different concentrations in Heliomolar Flow (Ivoclar Vivodent AG, FL-9494) resin composites promise future use of these structures as dental resin composites.

Nanotechnology as a novel technique has contributed to the use of nanoparticles in the organic resin matrix of dental composites at a nano-scale. In fact, due to the high surface to volume ratio of the nanoparticles, they can improve the mechanical, esthetic and optical properties as compared to those of the conventional, microfilled and hybrid resins.

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DISCLOSURE STATEMENT

All authors declare that they have no competing or no conflict of interests.

COMPLIANCE WITH ETHICS REQUIREMENTS

This article does not contain any studies with human or animal subjects.

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