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METHODS AND TECHNIQUES USED IN THE DEVELOPMENT OF CERAMIC-REINFORCED PHOTOPOLYMER COMPOSITES FOR 3D PRINTING

BY

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Abstract. Ceramic-reinforced photopolymer composites have attracted considerable attention for additive manufacturing applications in regenerative medicine, digital dentistry, and biomedical engineering. The incorporation of ceramic fillers into photocurable resin systems enables the fabrication of structures with enhanced mechanical properties, dimensional stability, bioactivity, and antimicrobial functionality. However, the successful processing of these materials by stereolithography (SLA), digital light processing (DLP), and LCD-based technologies requires careful control of suspension composition, rheological behavior, photopolymerization kinetics, and post-processing conditions. This paper reviews the principal methods and techniques employed in the development of ceramic-filled photopolymer composites. Particular attention is devoted to resin formulation, ceramic filler selection, suspension preparation, particle surface modification, and characterization techniques including SEM, EDS, XRD, UV-Vis spectroscopy, rheological analysis, thermal characterization, and mechanical testing. The relationships among particle size distribution, curing behavior, microstructure, and final properties are discussed. Current challenges and future

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research directions are also highlighted, emphasizing multifunctional and biocompatible materials for advanced biomedical applications.

Keywords: additive manufacturing; photopolymer composites; ceramic fillers; stereolithography; digital light processing; biomaterials.

1. Introduction

Additive Manufacturing (AM), commonly referred to as 3D printing, has emerged as one of the most transformative technologies in modern manufacturing due to its ability to fabricate complex structures directly from digital models while offering unprecedented geometric freedom, reduced material waste, and extensive customization capabilities. These advantages have facilitated its widespread adoption across diverse fields, including regenerative medicine, digital dentistry, biomedical engineering, aerospace applications, and the development of advanced smart biomaterials (Yared and Gadow, 2023).

Photopolymerization-based three-dimensional (3D) printing technologies, particularly Stereolithography (SLA) and Digital Light Processing (DLP), have emerged as some of the most advanced additive manufacturing approaches owing to their ability to fabricate geometrically complex structures with exceptional dimensional accuracy, high resolution, and unprecedented design flexibility (Lin *et al*., 2025). These technologies have attracted considerable attention in biomedical engineering, regenerative medicine, and digital dentistry, where the integration of bioactive ceramic fillers into photocurable polymer matrices has enabled the development of multifunctional biomaterials with tailored physicochemical and biological properties. Despite their significant potential, the fabrication of ceramic-filled photopolymer systems remains challenging due to the complex interactions occurring during the photopolymerization process (Bove *et al*., 2022). Overall, the additive manufacturing of ceramic-reinforced photopolymer composites represents a rapidly expanding field with significant implications for regenerative medicine, biomedical engineering, and digital dentistry (Hada *et al*., 2022). Despite considerable advances, several challenges remain, including the stabilization of highly loaded suspensions, mitigation of particle sedimentation, optimization of photopolymerization and post-processing conditions, and a comprehensive understanding of the relationships among material composition, microstructure evolution, and long-term biological performance. Current research efforts are therefore focused on the development of multifunctional, bioactive, and customizable composite materials capable of meeting the increasingly complex demands of personalized and digitally driven healthcare technologies.

In ceramic stereolithography, the efficiency of light-induced curing is strongly governed by the interplay between incident radiation, the organic resin matrix, and dispersed ceramic particles. Among the various parameters involved,

particle size distribution (PSD) plays a pivotal role in determining the optical behavior, rheological characteristics, and curing kinetics of the suspension. Fine particles tend to increase light scattering and attenuation, thereby reducing the penetration depth of ultraviolet (UV) radiation and limiting polymerization efficiency. Conversely, larger particles may improve suspension flowability and enhance curing rates, although they often increase the risk of sedimentation and phase separation. Consequently, the optimization of ceramic suspensions requires a careful balance between colloidal stability and processability in order to achieve reliable printing performance and high-quality components. Another important research direction involves optimizing the interfacial interactions between ceramic fillers and the organic polymer matrix through the use of silane coupling agents. Surface functionalization of ceramic particles with vinyltriethoxysilane (VTES) has been reported to improve filler dispersion and distribution within the resin system, promoting the formation of a more homogeneous interface and enhancing the mechanical properties of the resulting composites. Nevertheless, the mechanisms through which interfacial modifications simultaneously affect rheological behavior, photopolymerization kinetics, and biological performance remain only partially understood and require further investigation (Jeon *et al*., 2025).

Among the various AM technologies, vat photopolymerization, primarily represented by Stereolithography (SLA) and Digital Light Processing (DLP), stands out for its exceptional dimensional accuracy, high resolution, and superior surface quality. Both technologies rely on the selective curing of photocurable resins through ultraviolet (UV) irradiation. The principal distinction between them lies in the curing mechanism: SLA employs a focused laser beam to selectively polymerize the resin in a point-by-point manner, whereas DLP solidifies an entire layer simultaneously through digital image projection, resulting in significantly higher manufacturing speeds. Furthermore, recent advances in Liquid Crystal Display (LCD) and Digital Micromirror Device (DMD) technologies have contributed substantially to improving process resolution, reproducibility, and manufacturing efficiency (Hussain *et al*., 2024).

The incorporation of ceramic fillers into photocurable resin systems introduces significant challenges related to suspension stability, rheological behavior, and photopolymerization efficiency. The overall performance of these systems is strongly influenced by several interconnected parameters, including particle size distribution (PSD), filler loading, refractive index mismatch, and sedimentation stability. Ceramic particles affect the propagation of ultraviolet (UV) radiation within the resin, thereby influencing curing kinetics and the spatial distribution of polymerization. Moreover, increasing solid loading generally leads to higher viscosity, reduced flowability, and greater processing complexity, particularly in highly filled suspensions intended for structural applications (Yun *et al*., 2016).

Particle size distribution plays a critical role in determining both processability and the properties of the final component. Bimodal particle distributions have been shown to improve particle packing efficiency, enhance densification, and reduce residual porosity following sintering. However, particle agglomeration remains a major concern, as it can generate structural defects, induce stress concentrations, and ultimately compromise the mechanical integrity of the fabricated structures. Therefore, achieving an optimal balance between particle loading, dispersion quality, and suspension stability remains a key challenge in ceramic-filled photopolymer systems.

The final performance of additively manufactured ceramic-reinforced composites depends not only on the printing process itself but also on subsequent thermal post-processing stages, particularly debinding and sintering. These treatments govern densification mechanisms, pore evolution, dimensional shrinkage, and microstructural development, ultimately determining the structural integrity and functional performance of the final component. Consequently, precise control of thermal processing parameters is essential for minimizing defects and achieving optimal mechanical, physical, and biological properties.

Despite significant advances in recent years, the relationships among particle size distribution, light propagation, photopolymerization kinetics, microstructural evolution, and the resulting properties of ceramic-filled photopolymer composites remain insufficiently understood. The development of next-generation biomaterials capable of simultaneously combining high mechanical performance, bioactivity, antimicrobial functionality, and efficient printability requires an interdisciplinary approach integrating materials science, polymer chemistry, photopolymerization engineering, and biocompatibility assessment. From this perspective, the additive manufacturing of ceramic-reinforced photopolymer composites represents a highly promising research field with considerable potential for advancing personalized medicine, regenerative therapies, and the next generation of advanced biomedical materials.

2. Materials Used in Photopolymer Systems

The scientific literature highlights the extensive use of photocurable matrices based on acrylic and methacrylic monomers and oligomers in vat photopolymerization technologies, including Stereolithography (SLA), Digital Light Processing (DLP), and LCD-based 3D printing systems. Among the most commonly employed constituents are methyl methacrylate (MMA), urethane dimethacrylate (UDMA), trimethylolpropane triacrylate (TMPTA), triethylene glycol dimethacrylate (TEGDMA), and 2-hydroxyethyl methacrylate (HEMA).

UDMA is widely preferred due to its superior mechanical properties and reduced polymerization shrinkage, whereas TEGDMA and HEMA are commonly incorporated to regulate resin viscosity and enhance crosslinking

density. Photopolymerization is typically initiated using photoinitiators compatible with ultraviolet light-emitting diode (UV-LED) sources operating at wavelengths around 405 nm. Commonly employed photoinitiators include bisacylphosphine oxide derivatives and diphenyl(2,4,6-trimethylbenzoyl)phosphine oxide (TPO), which provide efficient radical generation and rapid curing kinetics.

To reinforce the polymer matrices, a variety of ceramic fillers have been investigated, including alumina (Al2O3), yttria-stabilized zirconia (Y-TZP, ZrO2-Y2O3), calcium carbonate (CaCO3), and bioactive ZnO/SiO2 systems. These materials are valued for their excellent mechanical performance, thermal stability, and biocompatibility, while ZnO/SiO2 particles additionally provide antimicrobial activity and enhanced biological functionality.

2.1. Preparation and Stabilization of Resin–Ceramic Suspensions

The preparation of stable resin–ceramic suspensions constitutes one of the most critical stages in the fabrication of ceramic-reinforced photopolymer composites. Current research focuses on powder conditioning procedures, the incorporation of dispersing agents and coupling agents, and the implementation of advanced homogenization techniques to minimize particle agglomeration and sedimentation.

Commercial dispersants such as Dolapix PC75, Dolapix CE64, Darvan-C, and DISPERBYK-2001 are frequently employed to improve colloidal stability and reduce interparticle interactions. Suspension homogenization is commonly achieved through mechanical stirring, ultrasonic treatment, and vacuum-assisted centrifugal mixing, followed by degassing procedures to eliminate entrapped air bubbles that may adversely affect photopolymerization efficiency and printing quality.

Numerous studies have investigated the influence of ceramic filler concentration on printability, rheological behavior, and the final properties of photopolymer composites. Consequently, considerable attention has been devoted to identifying an optimal balance between ceramic loading and processability in order to achieve both high-performance materials and reliable manufacturing conditions.

2.2. Characterization of Ceramic Fillers

The characterization of ceramic fillers is essential for understanding the behavior of photocurable suspensions and predicting the performance of the resulting composites. Particle size distribution (PSD) is commonly determined by laser diffraction techniques, whereas particle morphology and agglomeration phenomena are evaluated using optical microscopy and scanning electron microscopy (SEM).

Energy-dispersive X-ray spectroscopy (EDS) and X-ray diffraction (XRD) analyses are routinely employed to assess elemental composition and crystalline structure, respectively. In addition, dynamic light scattering (DLS) and zeta potential measurements are frequently used to evaluate the colloidal stability of ceramic suspensions. These characterization techniques provide valuable information regarding filler–matrix interactions and their influence on the rheological, mechanical, and functional properties of ceramic-reinforced photopolymer composites.

2.3. Optical Characterization and Photopolymerization Studies

The optical properties of ceramic-filled photopolymer suspensions play a fundamental role in determining the efficiency and accuracy of the photopolymerization process. Ultraviolet–visible (UV-Vis) spectrophotometry is commonly employed to evaluate light absorption, scattering, and transmission phenomena within the suspension. The interpretation of these optical characteristics is frequently based on the Beer–Lambert law, which provides valuable information regarding light attenuation and penetration depth during curing.

Photopolymerization kinetics are typically investigated using photorheological techniques and ultraviolet-assisted differential scanning calorimetry (photo-DSC). Key parameters, including gelation time, storage modulus (G') evolution, reaction enthalpy, and degree of conversion, are used to assess curing efficiency and to elucidate the influence of ceramic fillers on polymer network formation. These analyses provide critical insights into the relationship between suspension composition, curing behavior, and the resulting structural properties of the printed materials.

2.4. Rheological Characterization

Rheological behavior represents a critical factor governing the success of SLA and DLP manufacturing processes. Most ceramic-filled photopolymer suspensions exhibit non-Newtonian shear-thinning behavior, characterized by a progressive decrease in viscosity with increasing shear rate. Such behavior is advantageous for additive manufacturing, as it facilitates material flow during recoating while maintaining sufficient stability at rest.

Rheological characterization is commonly performed by measuring viscosity over a broad range of shear rates, allowing the evaluation of the effects of filler concentration, particle size distribution, and dispersing agents on suspension processability and stability. These investigations are essential for optimizing printing parameters and ensuring reproducible manufacturing performance.

2.5. Fabrication of Structures by 3D Printing

The majority of studies employ SLA, DLP, or LCD-based 3D printers equipped with ultraviolet light-emitting diode (UV-LED) sources operating at wavelengths of approximately 405 nm. Three-dimensional models are designed using computer-aided design (CAD) software and subsequently converted into STL files for layer-by-layer fabrication. Standardized specimens are typically produced to evaluate the mechanical, dimensional, and functional properties of the resulting materials.

Printing parameters, including layer thickness, exposure time, and irradiation intensity, are optimized according to the composition and rheological characteristics of the suspension. Following fabrication, printed specimens undergo cleaning procedures to remove uncured resin and are subsequently subjected to ultraviolet post-curing treatments to complete polymerization, enhance crosslinking density, and improve dimensional stability.

2.6. Microstructural and Thermal Characterization

Microstructural characterization is predominantly performed using scanning electron microscopy (SEM) coupled with energy-dispersive X-ray spectroscopy (EDS). These techniques are employed to evaluate filler distribution, interfacial interactions, and the presence of structural defects within the composite architecture. Such analyses provide valuable information regarding the homogeneity of the material and the effectiveness of particle dispersion within the polymer matrix. The main stages for preparation of photopolymerizable ceramic composite suspensions and 3D printing process and characterization methods and techniques are given in Fig. 1.

Thermal stability and residual ceramic content are commonly investigated using thermogravimetric analysis (TGA), which enables the assessment of degradation behavior, organic content removal, and thermal resistance. Together, these characterization methods facilitate the correlation between material composition, processing conditions, microstructural evolution, and the final performance of ceramic-reinforced photopolymer composites.

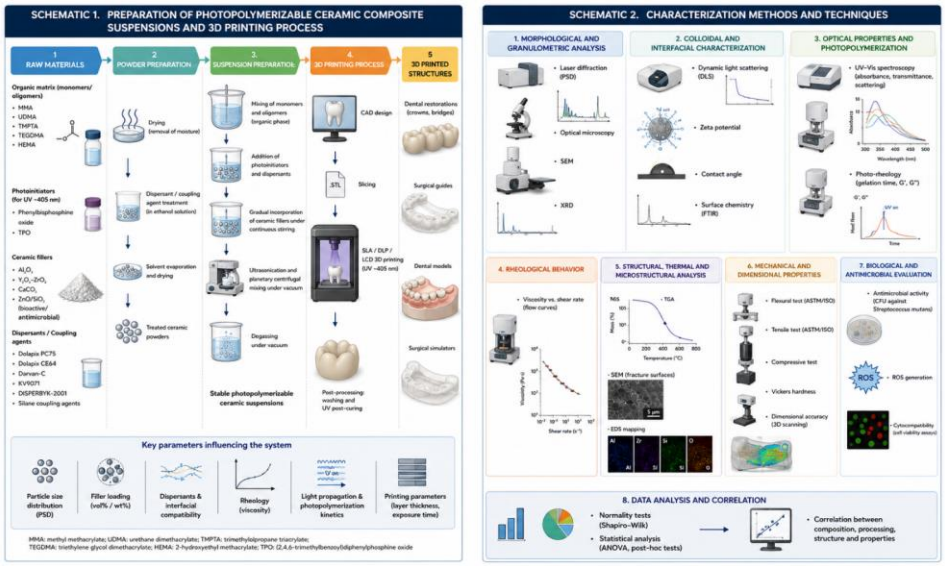


Fig. 1 – Preparation of photopolymerizable ceramic composite suspensions and 3D printing process and characterization methods and techniques.

2.7. Evaluation of Mechanical and Dimensional Properties

The mechanical performance of ceramic-reinforced photopolymer composites is commonly assessed through flexural, tensile, compressive, and hardness testing, conducted in accordance with relevant ASTM and ISO standards. The most frequently investigated parameters include flexural strength, elastic modulus, tensile strength, compressive strength, and Vickers hardness. These measurements provide essential information regarding the load-bearing capacity, stiffness, and overall mechanical reliability of the fabricated structures, particularly for biomedical and dental applications where long-term functional performance is required.

Dimensional accuracy is typically evaluated using three-dimensional scanning techniques and by comparing the geometry of the fabricated components with the original computer-aided design (CAD) model. Such analyses enable the quantification of dimensional deviations, shrinkage phenomena, and manufacturing accuracy, thereby providing valuable insights into the precision and reproducibility of the additive manufacturing process.

2.8. Evaluation of Biological and Antimicrobial Properties

For bioactive composite systems, biological performance is frequently assessed through antimicrobial investigations targeting Streptococcus mutans, one of the primary microorganisms associated with the development of dental

caries. Antimicrobial efficacy is commonly evaluated by quantifying colony-forming units (CFU) and by measuring the generation of reactive oxygen species (ROS), which are considered among the principal mechanisms responsible for the antibacterial activity of ZnO/SiO2-based systems.

In addition to antimicrobial testing, the biological potential of these materials is often examined through assessments of cell viability, cytocompatibility, and cellular response, providing a broader understanding of their suitability for biomedical and dental applications. Such evaluations are particularly important for the development of multifunctional biomaterials that combine mechanical reinforcement with biological activity.

3. Data Analysis and Interpretation of Results

Statistical analysis is generally performed using normality tests followed by parametric methods such as analysis of variance (ANOVA), complemented by appropriate post-hoc procedures for multiple comparisons. These statistical approaches enable the identification of significant relationships among material composition, rheological characteristics, photopolymerization behavior, mechanical performance, dimensional accuracy, and biological activity.

The integration of experimental data obtained from optical, rheological, mechanical, microstructural, thermal, and biological investigations contributes to a comprehensive understanding of the structure-process-property relationships governing ceramic-reinforced photopolymer composites. Such an integrated approach is essential for optimizing material formulations, improving manufacturing efficiency, and advancing the development of high-performance multifunctional biomaterials for personalized medicine, regenerative therapies, and digital dentistry.

Future developments in this field are expected to focus on:

- the formulation of advanced biocompatible photocurable resins;
- further optimization of DLP and LCD manufacturing technologies;
- the integration of digital simulation and predictive modeling tools for process control;
- the development of multifunctional and multi-material biomaterials;
- the fabrication of intelligent structures integrating mechanical, biological, and antimicrobial functionalities;
- the implementation of sustainable strategies for the preparation, processing, and recycling of photocurable resin systems.

4. Conclusions

Ceramic-reinforced photopolymer composites represent an important class of advanced materials for additive manufacturing technologies, particularly in biomedical engineering, regenerative medicine, and digital dentistry. The

performance of these materials is strongly influenced by the interactions among ceramic fillers, photocurable matrices, and processing parameters.

Particle size distribution, filler loading, suspension stability, and photopolymerization behavior were identified as critical factors governing printability, microstructural development, and the resulting mechanical and functional properties. The reviewed studies demonstrate that appropriate control of these parameters can significantly improve dimensional accuracy, mechanical strength, bioactivity, and antimicrobial performance.

Advanced characterization techniques, including SEM, EDS, XRD, UV-Vis spectroscopy, rheological measurements, thermal analysis, and mechanical testing, provide essential information for understanding structure-property relationships and optimizing material formulations.

Future research should focus on the development of highly biocompatible resin systems, multifunctional ceramic fillers, predictive process modelling, and sustainable manufacturing strategies. These advances are expected to facilitate the design of next-generation photopolymer composites for personalized medical and engineering applications.

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METODE ȘI TEHNICI UTILIZATE ÎN DEZVOLTAREA COMPOZITELOR FOTOPOLIMERIZABILE ARMATE CU PARTICULE CERAMICE PENTRU IMPRIMARE 3D

(Rezumat)

Compozitele fotopolimerizabile armate cu particule ceramice reprezintă o clasă modernă de materiale utilizate în fabricarea aditivă pentru aplicații biomedicale, stomatologice și inginerești. Integrarea particulelor ceramice în rășini fotopolimerizabile permite obținerea unor structuri cu proprietăți mecanice îmbunătățite, stabilitate dimensională, bioactivitate și funcționalități antimicrobiene. Lucrarea prezintă principalele metode și tehnici utilizate pentru dezvoltarea, procesarea și caracterizarea compozitelor fotopolimerizabile armate cu ceramică destinate tehnologiilor SLA, DLP și LCD. Sunt discutate materialele utilizate, prepararea și stabilizarea suspensiilor, caracterizarea optică, reologică, microstructurală, termică și mecanică, precum și evaluările biologice și antimicrobiene. De asemenea, sunt evidențiate principalele provocări asociate suspensiilor cu încărcare ceramică ridicată și direcțiile actuale de cercetare privind dezvoltarea biomaterialelor multifuncționale pentru medicina personalizată și stomatologia digitală.