



Research Article

Printability of zinc metal ink for porous lattice structure

Rupendra Singh TANWAR¹ , Suyog JHAVAR^{2,*}

¹Department of Mechanical Engineering, Institute of Engineering and Science Indore, IPS Academy, Indore, 452012, India

²School of Mechanical Engineering, VIT-AP University, Inavolu, Beside AP Secretariat Amaravati, Andhra Pradesh, 522237, India

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ABSTRACT

In response to the critical limitations of donor site availability and risks of harvesting morbidity associated with autologous bone grafts, as well as the potential risk of disease transmission from allografts, there is a significant demand for artificial bone scaffolds in bone tissue engineering. This study introduces a novel thermoset biopolymer binder, specifically formulated to enhance the printability and performance of zinc-based direct ink writing (DIW) scaffolds, distinguishing it from previously reported systems. Zinc scaffolds are extremely promising due to their excellent biocompatibility, degradability and the ability to mimic the microstructure and properties of human cancellous bone. This work systematically investigated the rheological behaviours and printability of a water-based ink consisted of polyvinyl alcohol (PVA) and polyethylene glycol (PEG) biodegradable polymers with zinc metal powder, which was the first attempt to fabricate advanced porous lattice structures for biomedical applications. The viscosity of the ink was 18,000 mPa·s, which decreased significantly to 500 mPa·s at a shear rate of 1000 s⁻¹, ensuring optimal flow behavior during extrusion. The research performed the combination of 10 mm/s print speed and 2 bar extrusion pressure was optimal for printing high-fidelity zinc lattice structures, using a nozzle diameter of 1.9 mm, extrusion pressures of 0.5, 1.5 and 2 bar and print speeds of 5 mm/s and 10 mm/s. This study demonstrates the potential for the use of a novel, rheologically optimized thermoset biopolymer binder in zinc-based DIW scaffolds that can serve as a structurally robust and biocompatible alternative to conventional bone grafts for bone tissue engineering.

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INTRODUCTION

Bone defects and fractures represent a significant global public health concern, arising from conditions such as osteoporosis, tumors, injuries, and orthopedic diseases. As a result, bone tissue is the second most transplanted tissue in

the world, with more than four million procedures per year involving bone transplantation and replacement materials [1]. Artificial bone implantation has attracted great attention of researchers. It is an important way to repair bone fractures and reduces the risk of disease transmission from the donor to the recipient. Cortical bone and cancellous

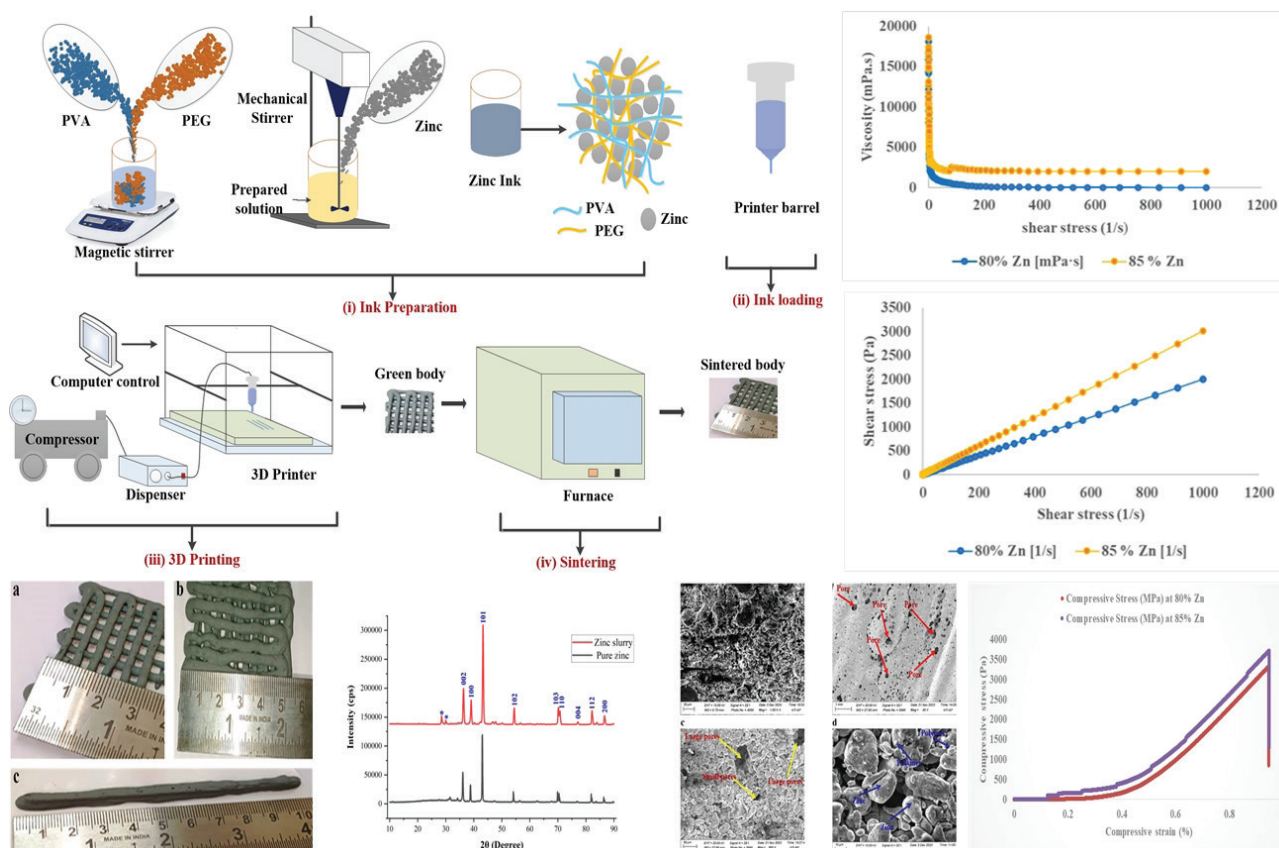
*Corresponding author.

*E-mail address: suyogjhavar@gmail.com

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GRAPHICAL ABSTRACT



bone are the two major types of hardened bone tissue, each serving a specific role in protecting internal organs and regulating metabolism. Cortical bone is dense and contains 5 to 10 percent air spaces. Its Young's modulus is 3 to 30 GPa. On the other hand, Cancellous bone, is highly porous (50-90% porosity) and consists of interconnected trabecular units, with a Young's modulus of 0.02-2 GPa. These features underline the interest of the cortical bone in the implantation of an artificial bone [2]. The mechanical and structural properties of artificial bone scaffolds should be similar to those of natural bone, allowing the free movement of cells and proteins. For these scaffolds to permit movement of interstitial fluids, e.g. blood and proteins, that are required for bone growth, they should have a similar Young's modulus and porosity to that of real bone. A too low Young's modulus can lead to mechanical failure and a too high one to stress shielding. Hence, the trade-off between structural porosity and Young's modulus should be carefully balanced as these properties are interdependent. Increasing one property usually results in decreasing the other property. The real challenge is to fabricate scaffolds with the required strength and the desired porosity [3][4].

Metallic implants are typically made from conventional non-degradable alloys such as stainless steel and titanium. But these materials often necessitate a second surgery to

remove and have risks of infection. Alternatives such as biodegradable metals like iron (Fe), magnesium (Mg) and zinc (Zn) alloys are being investigated. Among these, Zn-based implants are particularly promising due to the degradation rates matching with the bone tissue regeneration and no harmful gas generation unlike Mg-based implants degrading too fast and Fe-based implants hindering the bone healing [5][6]. Zinc (Zn) is a promising candidate for orthopedic implant materials because of its biodegradability, good mechanical properties, high biocompatibility, and osteopromotive effects. The geometry of the implant is also an important factor in the design of advanced bone replacements [7]. Recent studies [1] have demonstrated that zinc-based implants are more bioactive and less inflammatory as compared to magnesium or iron alloys further confirming its suitability for biomedical applications. To regenerate bone, porous structures are preferred because of the interconnected pore networks that facilitate cell proliferation. Additive manufacturing allows the precise design of porous structures and is therefore an ideal method for the fabrication of bone substitutes with complex shapes and increased bio-functionalized surface area [2][8]. While polymeric inks have been extensively used for DIW, zinc metal inks have been scarcely used for scaffolds. DIW is a cost-effective and flexible way to produce scaffolds with desired

porosity, allowing to control precisely the flow of material and the mechanical properties of the final scaffold. This highlights its significant potential for advancing zinc-based scaffold fabrication for biomedical applications.

Additive manufacturing (AM) stands out because it can create custom structures with intricate shapes and pores that can be adjusted. This is an ideal way for fabricating bone scaffolds with specific characteristics [9]. Alloys of zinc, titanium, magnesium and iron have been fabricated for bone implants by means of Laser Powder Bed Fusion (LPBF) and Direct Energy Deposition (DED). But zinc melts at a comparatively low temperature (420°C) and boils at 907°C. This makes it susceptible to oxidation and pore formation under high-energy laser or electron beams. Hence, LPBF and DED might not be the best techniques for the production of zinc alloy structures with desired porous properties [8] [10]. Direct Ink Writing (DIW) is a cheaper additive manufacturing process. It works by squeezing a paste through a small nozzle to print the object you want. This technique allows precise control of the material flow, which contributes to successful printing without any distortion of the shape [11]. DIW has been used for the fabrication of structures and films from polymer resins and is one of the leading AM techniques for printing of polymer composites [12]. The ink properties as well as the specific printing settings determine the strength and overall performance of DIW-fabricated parts. Ink properties include ink thickness or viscosity and the way ink reacts to shear stress or force. Printing settings are the size of the nozzle, the speed of its movement, and the pressure that is applied to push out the ink. These various properties are closely interrelated and influence each other, ultimately defining the characteristics of the final product. The research on the ease of printing with the polymer-based metal powder inks and their properties correlated with their structure is not abundant [13][14].

In this paper, we explore the use of zinc metal ink in additive manufacturing, in particular, in the DIW process. The zinc metal ink is a composite, water-based ink, which is formed by mixing zinc metal powder with biodegradable polyvinyl acetate (PVA) and polyethylene glycol (PEG). This ink has the right rheological properties for DIW and can form porous structures. Its purpose is to improve the printability of the ink and to adapt its physical and mechanical properties. This often means that scientists have to design ink recipes with multiple materials, which makes the system more complex. The printability of the prepared zinc metal ink can be evaluated by studying the flow behaviour of the ink. The microstructure and mechanical strength of the sintered samples are studied by X-ray diffraction (XRD), scanning electron microscopy (SEM) and compression tests.

The main innovations of this research are related to several aspects:

- **Heat-Free Fabrication:** a heat-free fabrication method, DIW, is applied in this work, which is advantageous for

temperature-sensitive materials because no external heat source is needed when fabricating the components.

- **Biodegradable Zn-Polymer Scaffolds:** research is aimed at developing biodegradable zinc-polymer scaffolds, designed with biodegradable polymers to ensure complete degradation of the final structure.
- **Optimized Ink Formulation:** A crucial aspect is the optimized ink formulation, achieved through a systematic approach of careful material selection and precise adjustments to the composition, aiming for optimal printability and structural strength.
- **Rheological Characterization:** The rheological properties of the ink are fully characterized to verify proper viscosity, shear-thinning behaviour and structural stability during the printing process.
- **Parametric Optimization:** Parametric optimization is another important aspect of this work. Critical printing parameters like extrusion pressure, nozzle speed and layer deposition strategy are carefully optimized to enhance the uniformity and structural accuracy of the scaffold.
- **Potential Biomedical Applications:** The scaffolds obtained in this work have promising properties for a variety of biomedical applications, especially for tissue engineering and development of biodegradable implants.

MATERIALS AND METHODS

The present study is aimed at the development of biodegradable scaffolds based on zinc and biodegradable polymers, that provide the printability of the final structure. This section details the materials employed for ink preparation, the stepwise procedure for ink formulation, rheological characterization, and parametric optimization for fabricating solid scaffolds.

Fabrication Strategy and Materials

The process parameters mentioned in the Table 1 and the fabrication process described in Figure 1, is composed of four steps: (i) ink preparation, (ii) ink loading, (iii) 3D printing and, (iv) sintering. The ink was composed Zinc metal powder of grain size is of 10 µm, and purity 98% (BFC, India), polyvinyl alcohol (PVA, molecular weight 350-400 g/mol, Eastchem India) and polyethylene glycol (PEG, molecular Weight, 1400-1600, shiv shakti group India) and Deionized (DI) water was utilized.

Ink Preparation

The optimal ink composition for direct ink writing (DIW) was established through a series of trials, as detailed in Table 2. Zinc granules with a solid content ranging from 80% to 85% by weight were identified as the most effective. At a weight percentage below 75%, it was observed that the printed layers were undesirably overlapped as seen in trials 1 to 4 of Table 2. The binder solution was prepared according

to the method of Elsayed et al. [2] and contained water, polyvinyl alcohol (PVA) and polyethylene glycol (PEG) in an approximate weight ratio of 73:17:10. To prepare the solution, the water was heated to 120 °C in a flask and then PVA and PEG were added. To ensure that all the reagents were dissolved completely, the mixture was homogenized at 600 rounds per minute with a magnetic stirrer for 90 minutes, as described in literature [2]. Afterward, the solution was allowed to cool to ambient temperature. The ink's pseudoplastic nature enables easy extrusion from the nozzles under low viscosity conditions (at high shear rates). It also helps stabilize the printed structures by significantly increasing viscosity at low shear rates. This pseudoplastic behaviour was achieved by incorporating PVA and PEG [15]. After that, zinc metal powder was slowly added to the aqueous solution and continuously mixed at 600 rpm for 60 min by mechanical stirring. After addition of all the powder, the zinc-water mixture was further agitated at a higher mechanical speed (1200 rpm for 20 min) to ensure thorough dispersion and to prevent from aggregates. This was a crucial step to avoid any interruptions during the 3D printing process. During mixing, ice was added to control the temperature and avoid overheating, which could cause loss of water or changes in viscosity through evaporation.

Rheological Characterization

The ink's flow behavior was assessed by conducting rheology measurements. Authors utilized a stress-controlled rotating rheometer (MCR102, modular compact rheometer, Austria). The experiments were conducted using a plate-plate configuration, with a diameter of 50mm and a gap of 1mm, at a temperature of around 25 °C. The parameters and procedure were determined based on the

information provided in the literature [16]. Firstly, the viscosity trend was measured as a function of the increase in the shear rate from 0.01 to 100 s⁻¹. The second measurement has to do with how long it took for the viscosity to recover. At first, a 20 second period was used with a constant shear rate of 1 s⁻¹. Overcoming the yield stress of the printing ink required a higher shear rate. Afterwards, a 450 Pa constant shear stress at the shear rate is 20. The second part of the test involved looking at how the recovery time trended for values of high viscosity. Thus, for the viscosity to recover in the second stage, the shear stress must be less than the yield stress. The shear stress drops as the shear rate goes up, which means the material is getting softer and able to print. As the shear rate increases, the viscosity of the shear-thinning fluid drops [17].

Direct Ink Writing of 3D Zn Scaffolds

Two designs were created and manufactured, featuring tetragonal lattices with orthogonal layers composed of 2 mm parallel struts and walls using inhouse developed 3D printer as shown in Figure 2. Both designs were fabricated using a layer height of 1 mm, determined through various trials to ensure good interfacial adhesion before drying after extrusion. For the production of Zn lattices with varying pore sizes and porosities, the X-Y spanning distances between the filaments were set at 2 mm, with a lattice length of 50 mm. Additionally, a wall with a height of 10 mm and a length of 100 mm was fabricated using the same zinc-based ink. To ensure the complete evaporation of water from the printed Zinc components, the scaffolds were left to air-dry overnight post-printing. Upon completion of printing and drying, the scaffolds underwent a sintering process as described in the literature. The process consisted of three

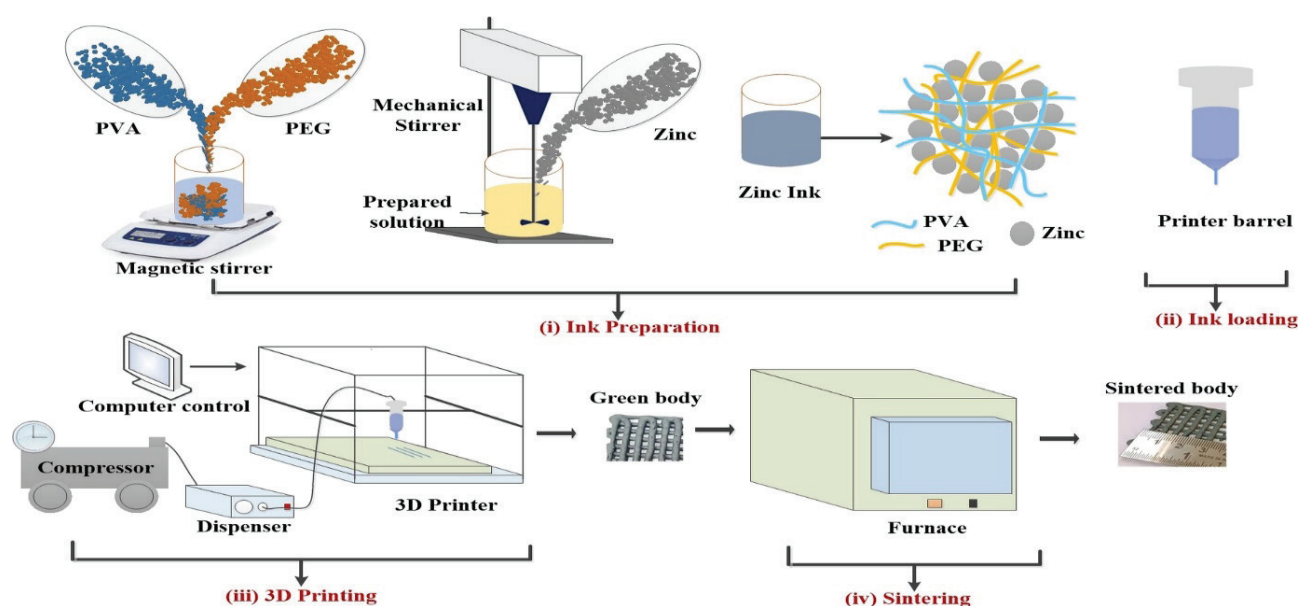


Figure 1. Zn lattice fabricate via direct ink writing steps.

Table 1. Process parameters

Parameters	Value
Syringe inner diameter	1.9mm
Speed of print head	10mm/sec
Pressure	2 bar
Zn metal powder Wt%	80%, 85%

stages: an initial plateau at 100°C for 30 minutes, followed by 150°C for 90 minutes, and finally, 200°C for 180 minutes, with a heating rate of 1°C per minute. The polymers were intended to be removed from the metallic particles during the initial stage at 100°C [18].

3D Printed Zn Part Characterization

The crystalline phase identification was performed using an X-ray powder diffractometer (Rigaku MiniFlex600-C, benchtop, 2.0 kW Cu X-ray tube, USA) on powdered samples from the 3D-printed Zn object and Zn metal powder. X-ray diffraction analysis was conducted with parameters set to 2θ ranging from 10° to 90°, with a step size of 0.05° and a duration of 5 seconds per step. The microstructural properties were examined using scanning electron microscopy (Zeiss EVO 10, USA). The mechanical properties of the as-constructed sample were initially assessed via a quasi-isostatic compression strength test. The experiment was carried out by Tinius Olsen H10KL Universal Compression Testing machine which can support a load up to 10 kN. The deformation rate was fixed at 0.2 mm/min. The mechanical robustness of the material was quantitatively determined by dividing the maximum load by the compression surface area to obtain the compression strength.

RESULTS AND DISCUSSION

3D-printed parts with complex designs and varying degrees of porosity can be fabricated using Direct Ink Writing (DIW), an additive manufacturing process that involves extruding thick slurries through a fine nozzle. A critical initial step in this process is developing an ink with controlled viscosity and appropriate rheological properties. The primary reason for using inks with the correct viscosity is to prevent filament distortion post-extrusion and ensure smooth flow through the thin nozzles utilized in the printing process. In this study, the addition of zinc metal powder to an aqueous solution containing polyvinyl alcohol (PVA) and polyethylene glycol (PEG) resulted in an ink with desirable rheological characteristics, as illustrated in Figure 3. This ink proved to be effective for the DIW process. The use of aqueous inks, even at low polymer concentrations, offers multiple benefits, including cost-effectiveness, ease of use, and non-toxicity. Moreover, aqueous inks facilitate precise drying, preventing slow rates that could compromise layer adhesion and lead to crack formation. They also enable burnout at lower temperatures.

The rheological characterization of the prepared ink, as depicted in Figure 3, demonstrates its inherent shear-thinning behaviour. The viscosity of the ink can be seen to decrease steadily with increasing shear rate from 18,000 mPa·s to 500 mPa·s at a shear rate of 1000 s⁻¹ (Fig. 3a). The specific shear-thinning behavior is a major advantage for direct ink writing (DIW) as it permits a continuous extrusion process that is needed for the layer-by-layer assembly of 3D printed structures. Furthermore, the inks exhibit pseudoplasticity, i.e., they show a yield stress, which is dealt with in Section 2.3. The lower viscosity of the ink at higher shear rates allows the ink to be extruded through small nozzles very efficiently allowing for high flow rates. Shear stress

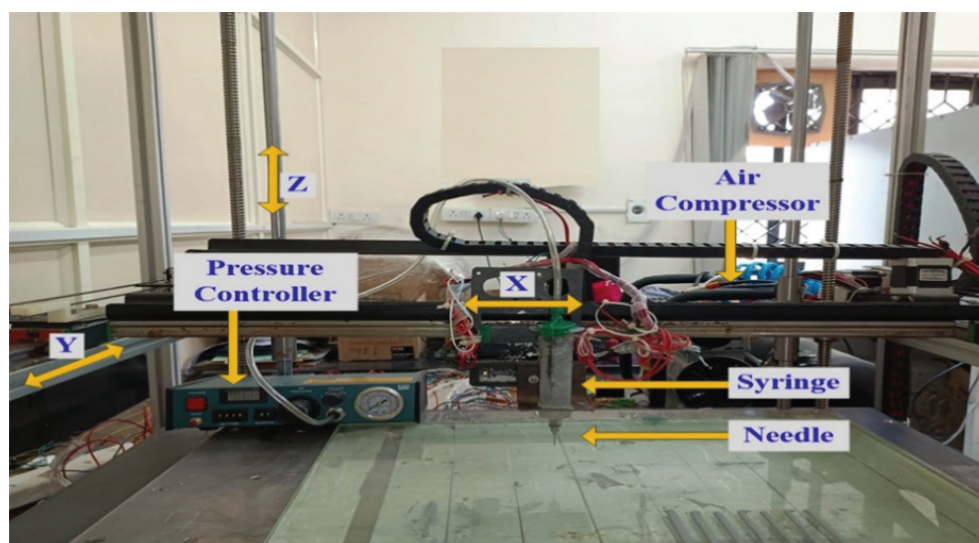
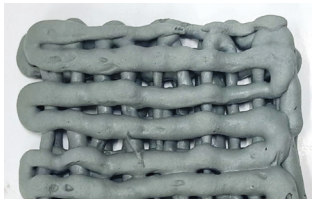
**Figure 2.** Modified 3D printer.

Table 2. Printing parameters summary

Trials	Syringe diameter (mm)	Speed of print head (mm/sec)	Pressure (bar)	Sample	Conclusion
1.	1.9mm	5	2		50 % Zn metal powder and very much flattened, no stacking at all, very poor stacking.
2.	1.9mm	5	0.5		60 % Zn metal powder and layers get mixed each other
3.	1.9mm	5	1.5		70 % Zn metal powder and layers not spreading evenly.
4.	1.9mm	10	0.5		75 % Zn metal powder and materials flow uneven.
5.	1.9mm	10	1.5		80 % Zn metal powder and mixed up structure
6.	1.9mm	10	2		85 % Zn metal powder and layers are stacked to each other and able to fabricate porous structure

increases linearly with shear rate with the maximum value up to 3000 Pa for Zn 85 % formulation. This behaviour indicates fast filament solidification and thus an increased structural stability, which is visually verified in Figure 4(a-c) for 3D-printed Zinc porous structures showing (a) a 0.8 mm spanning distance, (b) a 0.5 mm spanning distance and (c) a porous wall configuration. The rheological data and subsequent analysis of the samples together confirm the developed inks have the necessary attributes for successful applications of DIW. The ink's high consistency during extrusion through a 1.9 mm nozzle was sufficient to

maintain the flow without collapse under its own weight. Consequently, highly porous 3D-printed zinc components, adhering to the CAD model, were successfully produced. The dye used in this study is Reactive Orange 16. Reactive Orange 16 is an anionic dye and azo belongs to the class. In Table 1 its characteristics.

X-ray diffraction (XRD) was employed to identify the crystalline phases present in the zinc parts produced via Direct Ink Writing (DIW) and in the zinc metal powder. The primary crystalline peaks of zinc (JCPDS number 00-004-0784) are shown in Figure 5. The peaks observed

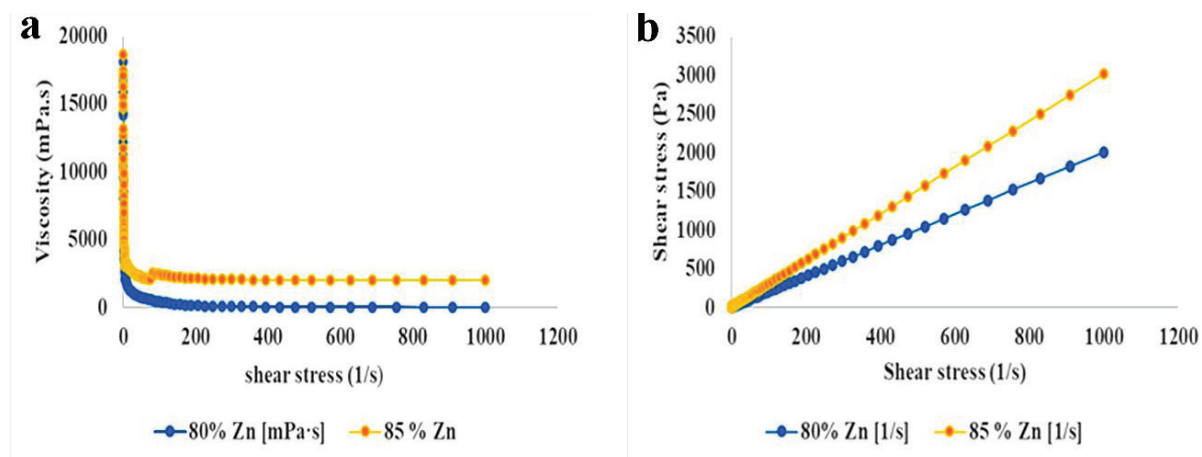


Figure 3. Rheological properties of prepared ink. a) indicate the viscosity function of shear rate b) ink shear stress with shear stress.

are consistent with the hexagonal close-packed (HCP) structure typical of zinc. Other peaks were observed due to the presence of polyvinyl alcohol (PVA) and polyethylene glycol (PEG) mixed in the zinc powder. The additional peaks (marked with *) are associated to the remaining polymeric phases as physical inclusions in the unsintered samples. These polymers act as binders and do not form chemical bonds to zinc. The XRD pattern of the zinc ink shows in particular two additional peaks, also marked with an asterisk (*), which are connected with the polymers

present in the zinc. Importantly, no significant secondary phases or impurities such as zinc oxide (ZnO) or other zinc compounds could be identified, confirming that the chosen processing conditions are effective in mitigating contamination. Apart from these additional signals, the most abundant peaks are those of zinc, which means that the zinc powder is the main component of the ink. Previous studies [4] have demonstrated the biocompatibility, sufficient strength, biodegradability, and cytocompatibility of zinc, all of which promote bone growth [4]. These results

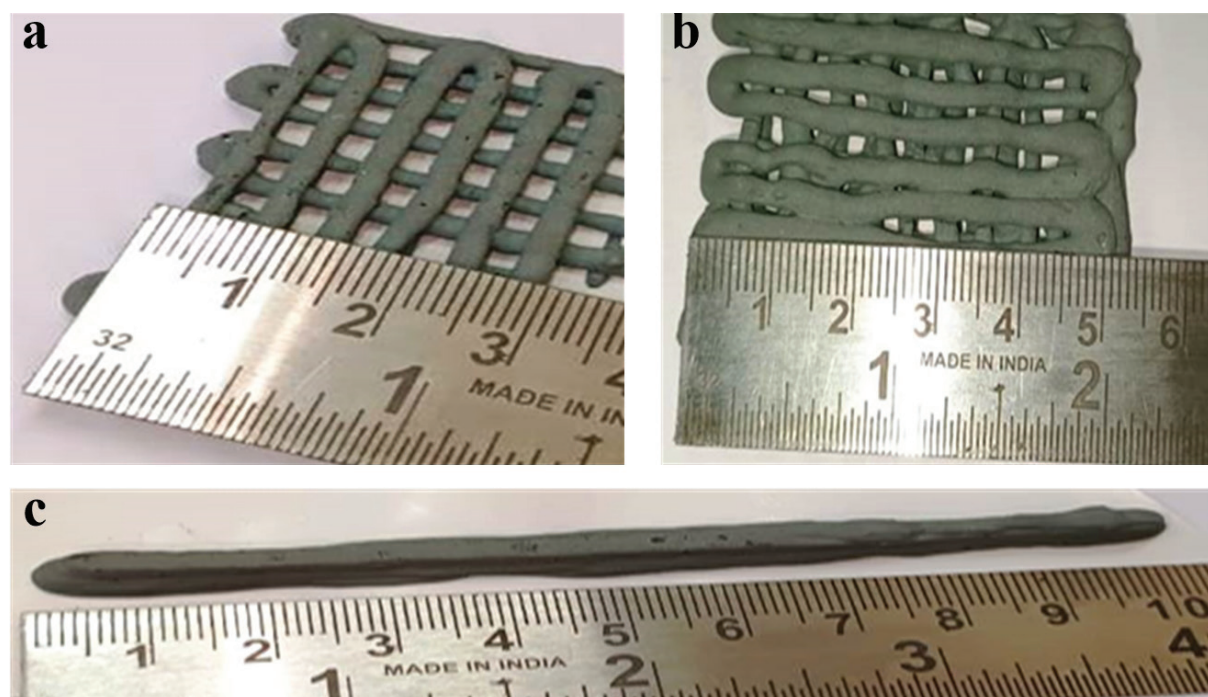


Figure 4. 3D printed Zinc porous structure a) 0.8 mm spanning distance, b) 0.5 mm spanning distance and c) wall with porosity.

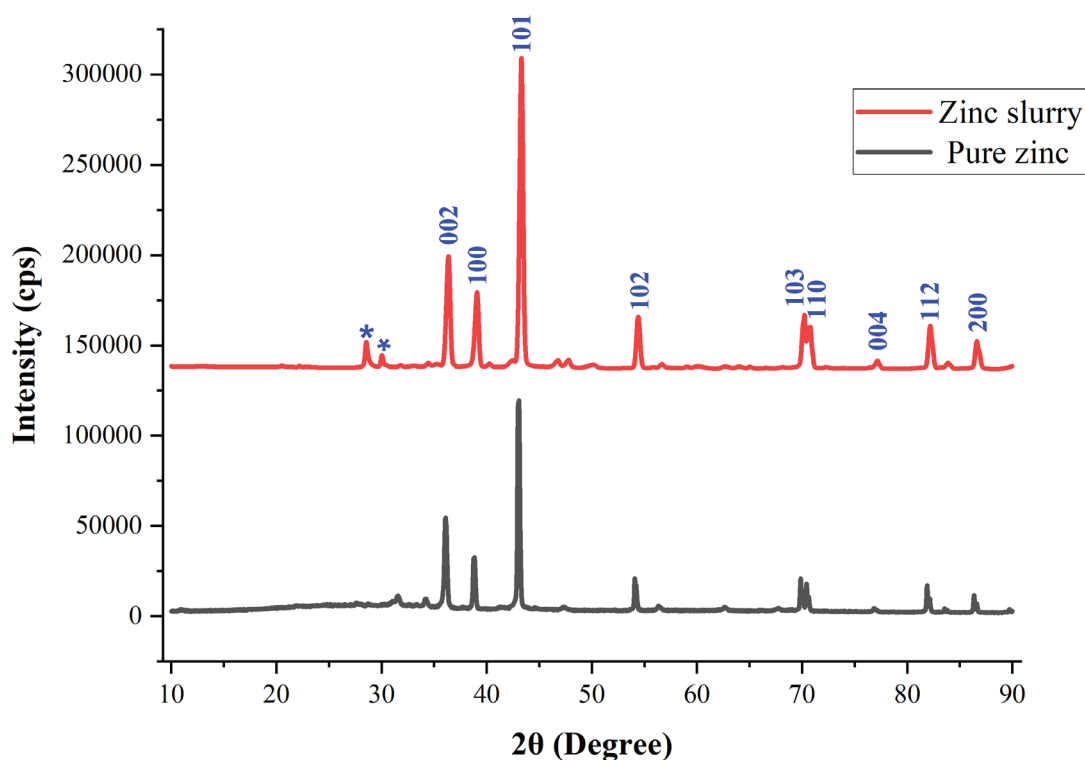


Figure 5. X-ray diffraction (XRD) patterns of pure Zn, Zn ink.

together confirm that Direct Ink Writing (DIW) is an effective method in fabricating pure zinc scaffolds for biomedical applications.

The microstructure of the Zn sample before and after sintering is shown in Fig. 6. Scanning electron microscopy (SEM) images show that the printed lattice and wall surfaces are smooth and free of cracks, with no sign of viscous collapse (Fig. 6a). The porosity of the structure is heterogeneous and consists of large and small pores (Fig. 6b-c). Quantitative analysis using ImageJ software shows the pore sizes in the range of 50 μm to 250 μm with an average pore size of 150 μm . At higher magnification, the microstructure exhibits a rough texture composed mainly of fine crystals (see Fig. 6d). The total porosity of the structure was found to be approximately 65%, which is in accordance with the requirements for biomedical scaffolds. Moreover, the average strut thickness was measured to be 300 ± 20 μm . These 3D-printed Zn supports exhibited minimal sagging, proper layer overlap, and diverse spanning structures. Upon examining SEM images of samples taken from a 3D zinc scaffold, which had been sintered at 200°C for four hours, we confirmed the successful fabrication. These images showed that the supports had minimal sagging, correct layer overlap, and diverse spanning structures. The individual filaments displayed only slight bending and deformation. It is important to note that the structure contained macrospores that matched the designed lattice, alongside intra-filament pores (Fig. 6a), which were probably created by air becoming

trapped during the ink preparation. Furthermore, these same intra-filament pores are also considered to be a consequence of gas entrapment during the de-binding process. Excellent adhesion was evident in the interlayer bonding, and no discontinuities were observed. It is also worth noting that small curvature radii within the scaffolds could potentially affect their mechanical strength. In summary, the SEM images confirmed the effectiveness of the DIW additive manufacturing method in creating porous structures, thus underscoring its broad potential across various applications.

When subjected to compressive loading, sintered scaffolds typically exhibit the behaviour shown in Figure 7, which illustrates the relationship between compressive stress and deformation. The densification of the cellular structure generally corresponds to a plateau region in the stress-strain curve; however, this behaviour is somewhat ambiguous in the current data. The loading process induces localized brittle fractures, particularly at the strut junctions, where stress concentrations lead to fracture formation. The four-hour sintering process at 200°C greatly contributes to the bonding of the particles and the densification of the materials, resulting in an improved adhesion of the layers and a marked reduction in porosity. These structural improvements are needed for the mechanical stability of the scaffolds. Comparing different ink compositions, scaffolds made with 85% zinc have a compressive yield stress of 3710 Pa, while those with 80% zinc have a lower value of

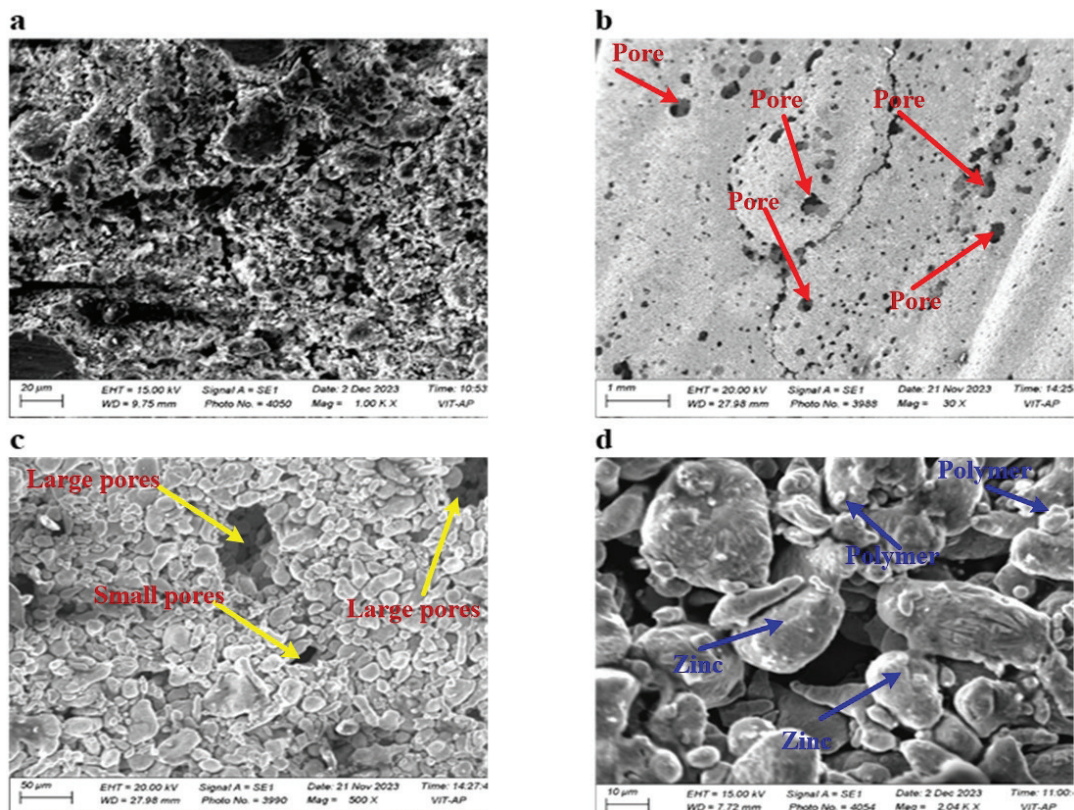


Figure 6. SEM images of Zn 3D printed scaffolds, a) at 20 μm , b) 1mm, c) 50 μm d) 10 μm .

3250 Pa. The two measurements were made at a compressive strain of 0.974 %. The X-ray Diffraction (XRD) analysis indicates that the main crystalline phases of zinc are not significantly changed after the sintering process, which suggests that the temperature used in the sintering process

is below the recrystallization temperature. However, in the Scanning Electron Microscopy (SEM) images, grain coarsening can be observed. This typically increases the load-bearing capacity of the material, but at the same time leads to a small decrease in yield stress, mostly because of

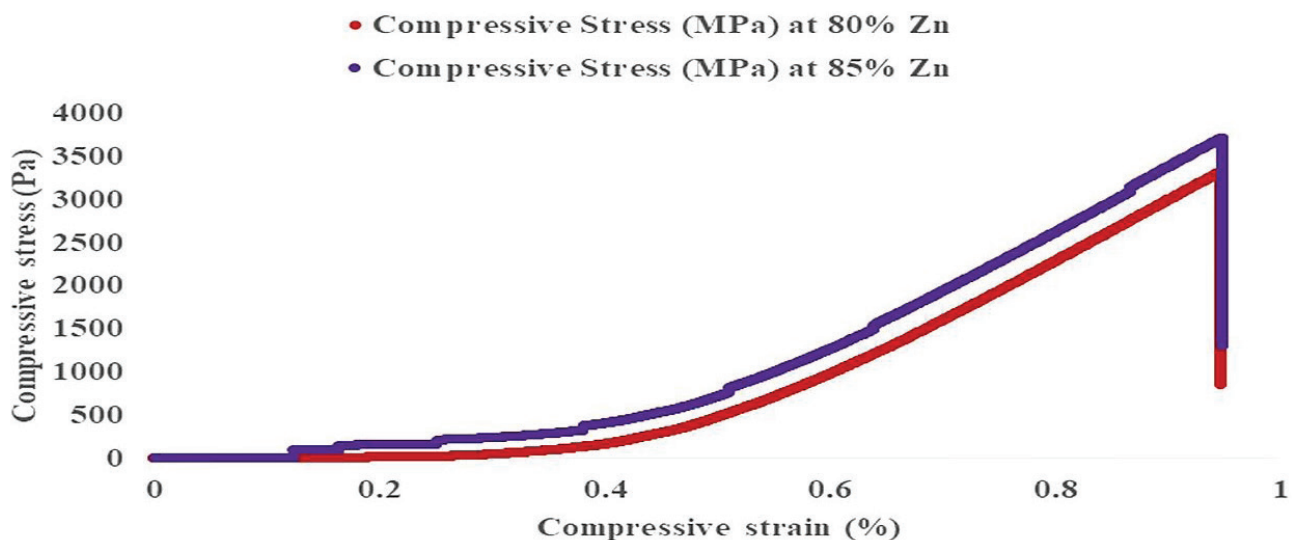


Figure 7. Compression strength DIW printed part.

reduction in grain boundary strengthening. The elastic modulus, obtained from the initial linear portion of the stress-strain curve was found to be 1.5 MPa for 85% zinc scaffolds and 1.2 MPa for 80% zinc scaffolds. The material shows a rapid drop in stress after the yield point which is a sign of brittle fracture. It is then followed by a progressive increase in stress due to the progressive densification of the porous internal structure. The results show that increasing the ratio of zinc powder is an effective way to improve the load capacity of zinc metal components. The stress-strain curve has a steep initial slope. The stress increases steeply as a function of the applied strain. However, it shows a sudden fall after reaching a certain value which is indicative of limited strain capacity of the fabricated sample. Moreover, the sintering process is able to efficiently remove any residual polymeric phases, thus assuring that the scaffolds are mainly composed of pure zinc. These additional metrics, including elastic modulus and post-yield behaviour, together provide a more comprehensive mechanical characterization of the sintered scaffolds.

CONCLUSION

This study demonstrates that the novel thermoset bio-polymer binder, combined with a zinc-based water-based ink, shows promising potential for creating porous lattice structures through Direct Ink Writing (DIW) for biomedical applications. This material shows considerable promise for creating artificial bone scaffolds. It represents a practical alternative to conventional bone grafts with improved biocompatibility and better structural properties. In conclusion:

1. The fabrication of zinc metal lattice structures and their surrounding walls has been successfully demonstrated in this research. The main solid component was zinc metal powder, and polyvinyl alcohol (PVA) and polyethylene glycol (PEG) were used in an aqueous solution.
2. As one of the key aspects of the study, the rheological properties of specially prepared ink were thoroughly analyzed. The aim was to obtain the optimal printing parameters for zinc structures, so that the manufactured dimensions were very close to the designed ones and the dimensional stability was very good. The results were verified by using both the scanning electron microscopy (SEM) and compression tests.
3. The best printing parameters for the zinc metal ink developed in this work were found to be a print head speed of 10 mm/s, an extrusion pressure of 2 bar and a solid loading of 85% zinc metal powder. The mixture was extruded through a nozzle of 1.9 mm diameter.
4. Specifically, the ink with 85 % zinc metal powder had a compressive stress of 3710 Pa at a compressive strain of 0.973 %.

This study has some limitations that should be noted, including the lack of in vivo testing and a comprehensive long-term biodegradability evaluation. The present findings

provide evidence for the potential of DIW-fabricated zinc scaffolds in biomedical applications and future research should ideally evaluate these scaffolds in vivo. This would require long-term in vivo studies to track their biodegradability and biocompatibility closely. Another promising direction for future work is to study the effects of different lattice geometries on mechanical properties and biological responses.

NOMENCLATURES

AM	Additive Manufacturing
DIW	Direct Ink Writing
PBF	Powder Bed Fusion
DED	Direct Energy Deposition
Zn	Zinc
SS	Stainless Steel
Mg	Magnesium
PVA	Polyvinyl Alcohol
PEG	Polyethylene Glycol

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AUTHORSHIP CONTRIBUTIONS

Authors equally contributed to this work.

DATA AVAILABILITY STATEMENT

The authors confirm that the data that supports the findings of this study are available within the article. Raw data that support the finding of this study are available from the corresponding author, upon reasonable request.

CONFLICT OF INTEREST

The author declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

ETHICS

There are no ethical issues with the publication of this manuscript.

STATEMENT ON THE USE OF ARTIFICIAL INTELLIGENCE

Artificial intelligence was not used in the preparation of the article.

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