

FDM Printing of Aluminum Feedstocks with focus on the debinding process

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Abstract

This study investigates the printability, debinding, and sinterability of aluminum-based (6000 series) feedstocks prepared with different commercial thermoplastic binder compositions, with a focus on the effect of the post-processing on microstructure and densification during sintering. Two debinding approaches were studied: (i) solvent debinding followed by thermal debinding, and (ii) single-step wick debinding, in which the green parts were embedded in a powder bed. Debinding is the most challenging stage for Al-containing feedstock. The high tendency of aluminum to oxidize significantly limits the choice of solvent and the debinding process in an oxygen atmosphere. To avoid oxidation of aluminum powder, the solvent spiece and debinding temperature in air must be restricted. This limitation can leave residues of carbon in the structure during the sintering in nitrogen atmosphere. Based on simultaneous thermal gravimetric analysis, the onset oxidation temperature for the 6061 aluminum powder used in this study was determined to be approximately 400°C; therefore, the thermal debinding temperature under air was selected below this limit. The results showed that with both debinding methods binder removal of $\geq 96\%$ could be achieved. However, optical microscopy and density measurements after sintering indicated that even high-purity acetone as a solvent medium resulted in slight oxidation, particularly at the printed sample surfaces. Interestingly, wick debinding without previous solvent debinding resulted in defect-free samples with higher post-sintering density. The effect of sintering dwell time was also examined. Increasing the holding time from 2 h to 10 h improved the density from $\sim 97\%$ to $\sim 98\%$.

1. Introduction

Aluminum alloys, particularly the 6061 alloy, have attracted significant attention due to their excellent strength-to-weight ratio, corrosion resistance, and various applications in industries such as aerospace, medicine, and automotive. Traditionally, manufacturing processes like casting, forging, and rolling have been used to shape aluminum and its alloys. However, these conventional methods offer limited design flexibility. While they are well-suited for producing larger components, they often struggle to meet the demands of advanced applications that require small, intricate, and highly precise geometries. In contrast, metal injection molding (MIM) of aluminum alloys excels in this regard, enabling the efficient production of small, complex parts with high dimensional accuracy and consistency. This makes MIM a more suitable choice for such specialized applications [1, 2]. The MIM process delivers precision, repeatability, speed, cost-efficiency, and excellent surface finishing with dense final parts. Liu et al. [3], developed an aluminum MIM feedstock based on AA6061 alloy mixed with tin, aiming to produce parts with mechanical properties comparable to conventionally processed alloy. They used a powder loading of 62 vol.% and a thermoplastic binder system consisting of stearic acid, palm oil wax, and high-density polyethylene. The injected parts underwent a two-step debinding process, including solvent debinding in hexane followed by thermal debinding, which was combined with sintering in a single furnace cycle. Using this approach, a relative density of 97% was achieved by sintering in a nitrogen atmosphere. Acar et al. [4], investigated the microstructural and mechanical properties of injection-molded aluminum powder. The feedstock with a powder loading of 62.5 vol.% and underwent solvent in heptane and

thermal debinding before sintering at various temperatures and durations in high-purity nitrogen. The maximum relative density obtained was 96.2% when sintered at 650°C for 60 min. These studies demonstrate that by careful control of powder loading, binder composition, and debinding/sintering conditions, it is possible to achieve high-density aluminum parts via MIM. However, the high cost of custom dies makes MIM process less economical for complex, low-volume parts [2]. In contrast, additive manufacturing (AM) technologies have gained widespread attention, particularly for metallic materials, owing to larger flexibility and cost-effectiveness for producing intricate or low-volume metal parts. Techniques such as vat photopolymerization, binder jetting, and directed energy deposition (DED) have been applied to fabricate parts from various metals, including steel [5, 6], titanium [7], copper [8, 9], and Ni-based superalloys [10]. Powder bed AM technologies, such as selective laser sintering (SLS) and selective laser melting (SLM), are among the most widely used for the fabrication of low-volume complex metal parts. Nevertheless, the use of direct energy sources like lasers or electron beams leads to extremely rapid solidification rates, up to ten times faster than traditional casting. These rapid solidification conditions limit the range of printable metal alloys to those that are easily weldable, restricting the adoption of high-performance alloys such as 6000- and 7000-series aluminum [11]. While laser powder bed fusion (LPBF) is widely used to process aluminum-based powders, this technique presents several challenges and often requires expensive equipment, inert atmospheres, and complex process control. These challenges arise primarily from aluminum's susceptibility to oxidation, the high energy required for melting, and its high thermal conductivity [12]. Early research into aluminum additive manufacturing, such as the work by Orme et al. [13] using direct droplet deposition, resulted in coarse-grained structures surrounded by significant oxide layers. Similar oxidation and microstructural issues were reported for selective laser melting (SLM) by Louvis et al. [14]. As a more accessible alternative, material extrusion-based additive manufacturing (MEX-AM), also known as Fused Deposition Modeling (FDM), has emerged as a promising method for producing metal parts using composite filaments. FDM printing reduces the requirement for handling loose metal powders, as printing of green parts is done exclusively with feedstock in the form of filament or granulate and there is no usage of powder beds. MEX-AM builds parts layer by layer through the extrusion of a thermoplastic-based feedstock and is popular due to its simplicity and low cost. Additionally, MEX-AM allows for the fabrication of multi-material components and functionally graded materials. However, challenges remain in achieving high mechanical properties and surface finish, especially when using metal-polymer composite feedstocks.

Malone et al. [15] investigated the application of FDM for fabricating aluminum 6061 parts using in-house formulated filaments. Following sintering, the maximum density obtained was around 70%, accompanied by relatively low hardness. The findings highlight the importance of binder selection and green-stage processing conditions, as inappropriate choices at this stage can significantly hinder densification. Momeni et al. [16], investigated Al feedstocks for FFF with different PP-based binder systems. They found that TPE/PPMA and TPE/PP/PPMA wax binders showed better rheological behavior than TPE/PP, though the latter exhibited greater ductility. However, they did not examine the debinding and sintering behavior. Manchili et al. [2] investigated the application of Al6061 metal injection molding granules in extrusion-based 3D printing, focusing on process performance and part quality

improvements. The primary objectives were to reduce surface roughness and improve the green density of the printed parts. A two-step solvent and thermal debinding process was applied prior to sintering. Solvent debinding was performed in acetone and thermal debinding, followed by sintering, was carried out under a pure nitrogen atmosphere. Based on SEM observations, two distinct regions were identified: a dense region at the center and a surrounding region that was not fully densified. Therefore, a critical step in the MEX-AM process is the development and selection of an appropriate thermoplastic binder system. The binder must be completely removable during debinding, leaving minimal carbon residue to ensure a high-purity metallic part. Complete binder removal typically requires a multi-stage debinding process, combining solvent and thermal debinding, and is often followed by sintering. As a result, thermal debinding and subsequent sintering of metallic parts produced by MIM or MEX-AM are generally performed in inert or reducing atmospheres. For aluminum-based systems, nitrogen atmospheres are commonly employed to limit oxidation [3, 4, 17]. However, the reliance on atmosphere-controlled furnaces, together with the extended cycle times required for thermal debinding, leads to high operating costs, representing a significant economic drawback. Aluminum-based feedstocks encounter particular challenges during debinding due to the high reactivity of aluminum powder, which increases the risk of oxidation during solvent and thermal debinding in air. Consequently, the use of air as the atmosphere for thermal debinding necessitates a binder system that is effectively removable at low temperatures, thereby minimizing oxidation during partial thermal debinding.

In this study, we investigate the 3D printing of aluminum powder using various commercial polymer binders via the MEX-AM (FDM) method. The research focuses on evaluating three critical stages of the process: (1) the printability Al-feedstock using standard FDM equipment with pellet extruder, (2) the debinding behavior of the polymer binder and its effect on the structural integrity of the printed parts, and (3) the sinterability of the green parts to achieve densification. This work mainly aims to advance the debinding process to be able to develop a low-cost, additive manufacturing method for high reactive metals. Using free kinetic modeling, we demonstrate both the feasibility and limitations of cheap 3D printed aluminum powder feedstocks based on commercial binder systems, with a special focus on the thermal debinding process in oxygen.

2. Materials and methods

2.1 Feedstock preparation:

The feedstocks were prepared by mixing pre-alloyed aluminum 6061 powder (D50: 31.9 μm , Ecka granules, Kymera international, Germany) with three different commercial thermoplastic binders: EnCeram (Chemische Fabrik Budenheim KG), Kcmix3.3 (later called Kcmix), and EmbemouldCC (KRAHN Chemie GmbH, Germany). The chemical composition of the aluminum powder is presented in Table 1.

Table 1
Chemical composition of the
aluminum powder obtained from the
manufacturer's safety data sheet (SDS)
[18]

Composition (wt.%)				
Al	Mg	Si	Cu	Cr
97.9	1.00	0.60	0.28	0.20

Density measurements of the aluminum powder and commercial binders were conducted using a helium pycnometer (Ultracyc 500, Anton Paar GmbH, Austria). The values were used to adjust constant filling volume (e.g. 48.3 cm³) of the torque rheometer. A torque Rheometer equipped with roller blade-shaped rotors (HAAKE PolyLab Rheomix 600, Thermo Fisher Scientific, Germany) with a rotational speed of 60 rpm for 30 min was used for the compounding process. The mixing temperature was adjusted according to the melting point of the commercial binders. The torque value during the mixing process was recorded to evaluate the prepared feedstock's homogeneity. Subsequently, all feedstocks were extruded through a nozzle with a diameter of 0.5 and 1 mm and a diameter ratio of 16 mm using a capillary rheometer (RH7 Flowmaster, Netzsch, Germany) and pelletized by a hand blender (KOENIG, Stabmixer Steel Line, Germany).

The optimization of powder content and determination of optimal solid loading were initially conducted using the EnCeram binder system as a reference. Feedstocks with progressively increasing aluminum powder volume fractions were prepared under a constant filling volume within the torque rheometer to identify the processing window and establish the target powder loading. Subsequently, feedstocks containing the determined powder volume fraction were formulated using three commercial binder systems; EnCeram, Kcmix, and EmbemouldCC, to comparatively evaluate their processability. The binder systems were assessed based on mixing behavior, as characterized by torque evolution during compounding, and extrusion stability during material extrusion. Binder systems that demonstrated stable compounding and consistent flow behavior were selected for further investigation, including printing, debinding, and sintering experiments.

2.2 Printing:

A screw-based FDM printer (Tumaker Voladora NX+, IT3D group, Spain) was used to print. Based on the results, printing was carried out using two feedstocks with a filler content of 60 vol.% aluminum powder prepared with EnCeram and Kcmix binders. Simplify3D software was employed for slicing the structures and generating the G-code files. The printing temperature was selected based on the melting points of binders. The corresponding printing parameters after optimization of the 3D shaping process are summarized in Table 2.

Table 2
Printing parameters used for the printing of disk structures of Al-feedstock with two commercial binder compositions (EnCeram and Kcmix, 60 vol.% of solid loading)

Printing parameters	Al-EnCeram	Al-Kcmix
Nozzle temperature (°C)	185	140
Nozzle diameter (mm)	0.8	0.8
Bed temperature (°C)	70	70
Printing speed (mm/s)	20	20
Extrusion width (mm)	0.8	0.85
Layer height (mm)	0.2	0.3
Flow (-)	2.7–5.4	2.7–3.9

2.3 Debinding and sintering:

To determine an appropriate debinding process for the prepared feedstocks, various approaches were implemented including solvent debinding followed by thermal debinding or by single-step of wick debinding before final debinding and sintering. Due to different binder compositions, different solvents are recommended to be used according to the manufacturers' guidelines. For the Al-EnCeram printed samples, water was used as a solvent and debound for 48 h at room temperature. The debinding behavior of the Al-Kcmix printed part was studied in acetone at 40°C for 24 h. A magnetic stirrer rotating at 150 rpm was used during this step. After the solvent debinding, all samples were dried at 40°C in air for 24 h. Thermal debinding for all samples with different binders was conducted in a box furnace (PC12 furnace, Pyrotek GmbH, Germany) under static air conditions for 24h. Although a debinding program up to 500°C is typically recommended for these commercial binders, thermal debinding in this study was limited to temperatures below the onset of oxidation because of the high reactivity of the aluminum powder (Fig. 1b).

For the single-step wick debinding process, the samples were placed within a highly porous Al₂O₃ powder bed (Nabalox NO 201, Nabaltec AG, Germany) and debound for 24h under static air conditions. For the final sintering, the samples were placed in a tube furnace (CTF 17/300, Carbolite, Germany) under nitrogen atmosphere with a continuous gas flow of 6.6 l/h up to 635°C with a constant heating rate of 5°C/min. The weight and the dimensions of the samples were measured and recorded before and after each step to assess the degree of polymer removal and the shrinkage behavior of the samples.

2.4 Characterization:

The crystallographic and phase structures of the Al powder were studied by X-ray diffraction (XRD, Xpert pro MPD, Malvern Panalytical Ltd, Germany) using Cu K α (λ = 1.5406 Å) radiation in the range of 20–90°

angles. The differential thermal analysis and thermogravimetric (DTA-TG, Jupiter F3 STA 449, NETZSCH, Germany) were employed to study the oxidation of the Al powder, melting point of binder components, and the binder burn-out behavior of samples in green and solvent debound states with 70 ml/min air flow and heating rate of 5 K/min. To study the kinetic of binder decomposition and the conversion rate during thermal debinding, kinetic analysis was conducted using the model-free Friedman method. For this mean, TG measurements with four different heating rates (1, 5, 10, 15 K/min) up to 400°C were carried out in air and the results were analyzed using Neo Kinetic software (Netzsch, Germany).

To evaluate the quality of the printed disk, the cross-section was examined using an optical microscope (ZEISS SteREO Discovery.V20, Carl Zeiss Microscopy GmbH, Germany) in both green and sintered states. The microstructure of the aluminum powder was characterized using a scanning electron microscope (SEM, VEGA3, Tescan, Czech Republic).

The rheological behavior of the feedstocks was studied on a rotational viscometer (MCR 302, Anton Paar, Austria) using a plate-plate setup with a 10 mm diameter and a gap of 1 mm. To ensure the reproducibility of the rheological measurements, for each feedstock two samples were measured and values of the second measurement are reported.

3. Results and discussion

3.1 Material characterization

Figure 1a presents the X-ray diffraction pattern of the as-received Al powder. Distinct diffraction peaks appear at 2θ values of approximately 38° and 45°, corresponding to the (111) and (200) planes of face-centered cubic aluminum (JCPDS File No. 004-0787). These results confirm that the powder is predominantly composed of elemental aluminum, with negligible contributions from alloying elements [19]. The morphology of the aluminum powder was examined using SEM. As shown in the inset of Fig. 1a, the Al particles are predominantly spherical, with a broad particle size distribution which is the characteristic of gas-atomized metal powders. It is well established in the literature that spherical powders exhibit superior packing density and flowability compared to irregularly shaped powders [20]. The surface morphology displays a generally smooth appearance with an orange-skin-like texture [21].

Because of its high reactivity, aluminum powder readily oxidizes upon exposure to oxygen, forming a passive oxide layer on the surface [22]. The presence of this oxide layer is known to be detrimental to the sintering process [38]. In contrast, oxygen plays a vital role during the removal and decomposition of the polymeric binder. To determine the onset temperature of Al oxidation, TG analysis was performed up to 600°C, i.e., below the melting point of Al (~ 660°C), under a synthetic air flow. The powders exhibit an exothermic peak between 450°C and 575°C, corresponding to Al oxidation. As shown in Fig. 1b, the first signs of oxidation were detected at temperatures between 400 and 450°C. Based on these findings, it can be concluded that Al6061 alloy powder should not be subjected to heat treatments exceeding 400°C in an oxygen-containing atmosphere.

DSC–TG analyses were carried out on three different commercial binder compositions to determine the binders melting point and decomposition behavior (Fig. 2). As shown in Fig. 2a and c, EnCeram and EmbemouldCC (EmbeCC) each consist of at least two components, showing melting points at approximately 63°C and 104°C for EnCeram, and 65°C and 111°C for EmbeCC. In contrast, the Kcmix binder exhibits a single primary melting transition at 62°C (Fig. 2b). The onset of thermal degradation was observed at 190°C, 140°C, and 210°C for EnCeram, Kcmix, and EmbeCC, respectively. The processing temperature window for each binder system lies between the melting point and the onset of thermal degradation.

3.2 Processability of the prepared feedstocks:

Before feedstock preparation, the density of the commercial binders and the aluminum powder was investigated. For the aluminum powder a density of $2.69 \pm 0.02 \text{ g/cm}^3$ was recorded. The EnCeram, Kcmix and EmbeCC binder exhibited densities of 1.09 ± 0.04 , 1.05 ± 0.05 and $1.07 \pm 0.03 \text{ g/cm}^3$, respectively. To find the optimum solid loading of aluminum powder in the feedstock, solid loadings ranging from 48 to 70 vol.% were tested while the mixing torque was studied. These investigations were carried out for the feedstock based on EnCeram binder. Figure 3 shows the relationship between the mixing torque and the solid loading. The torque increased nearly linearly with solid loading; therefore, identifying an optimum solid loading based solely on torque measurements was not feasible. Consequently, based on literature reports indicating a typical solid loading of approximately 62 vol.% for aluminum-filled feedstocks in MIM processes, a solid loading of 60 vol.% was selected in this study to avoid excessive torque and reduce the mechanical load on the printer head [3].

Using 60 vol.% as the solid loading for further investigations, mixing was carried out with various binder compositions. The equilibrium torque values at the end of the mixing process for feedstocks containing different binder compositions are shown in Fig. 4a. No significant changes in torque were observed, as the variation was within a tolerance of approximately ± 0.2 in the mean torque values. Minor differences can be attributed to variations in the binder compositions. Although the decomposition behaviors of the three different binder compositions were distinctly different (e.g. indicating variations in their components) the mixing results suggest that they contain components with similar flow behavior.

The flow behavior of the prepared feedstocks was investigated using a rotational plate-plate rheometer setup, operating at shear rates ranging between 0.001 and 0.1 s^{-1} to prevent the feedstocks from escaping the shear gap. Figure 4b presents the results of the rheological measurements conducted on prepared feedstocks with different binder compositions. Due to their different processing window, the tests were carried out at different temperatures. For all feedstocks, the measurement temperatures were set slightly below their respective onset decomposition temperatures. The chosen temperatures were 130°C for Al-Kcmix and 180°C for Al-EnCeram and Al-EmbeCC to enable direct comparison of their rheological behavior. Although both feedstocks show relatively similar flow behavior at higher shear rates, which confirms the results from the high shear compounding studies, at lower shear rates, the Al-EnCeram shows a stronger yield-point behavior. As shown in Fig. 4b, all feedstocks exhibited a decrease

in viscosity with an increasing shear rate, indicating a shear-thinning behavior. This rheological characteristic is advantageous for FDM processing, as it facilitates smoother extrusion through the nozzle while maintaining sufficient viscosity at rest to retain the shape and stability of the printed layers [23].

Figure 4c presents the pressure changes recorded during extrusion of the prepared feedstocks. As with the rheological measurements, the extrusion temperature was adjusted according to the binder type. The primary purpose of extrusion was to fabricate filaments, which were subsequently cut into pellets for the printing process. By continuously measuring the pressure during this step, feedstock homogeneity can be assessed through pressure fluctuations. For feedstock containing the EnCeram binder, an extrusion temperature of 160°C was applied, while for the other two binder systems, a lower temperature of 140°C was selected. Aside from the different pressure levels, the Al-EnCeram and Al-Kcmix feedstocks exhibit a similar pressure profile. Following a sharp initial increase, the pressure stabilizes and reaches a steady plateau in both materials (Fig. 4c). The Al-EnCeram Feedstock was extruded at significantly higher temperatures to achieve a filament with a smooth surface. Therefore, the pressure value of the plateau is much lower in comparison to the Al-Kcmix feedstock. In contrast, the Al-EmbeCC feedstock showed a continuous increase in pressure throughout the process. The presence of peaks in the pressure curve indicates feedstock inhomogeneity. The pressure increase is related to the binder separation, which was visually confirmed during the extrusion process. This phenomenon, which can be attributed to an insufficient interaction between polymer and metal powder, has been reported previously [24]. Such phase separation can negatively affect the 3D printing process by causing extrusion instabilities and poor layer adhesion [25]. Due to observed phase separation, further printability investigations of the Al-EmbeCC feedstock have not been pursued.

3.3 3D printing:

To enable printing with the prepared feedstock, a series of parameters were tested. One key factor investigated was the effect of temperature on the printing process. As illustrated in Fig. 5a,b, in a screw-based extruder, there are three different zones, including feeding, compression and melting zone. Temperature plays a crucial role in feedstock flow and extrusion stability. Excessively high temperatures in the feeding zone can lead to unwanted softening or even partial melting of the feedstock, causing granulate accumulation and thus feeding issues. Conversely, in the compression zone, the feedstock must remain partially solid in the upper section to provide sufficient compressive force, while controlled softening in the middle region ensures smooth extrusion. A broad temperature range of 110–185°C was investigated for the melting zone during printing with Al-EnCeram. Higher printing temperatures facilitated improved flow through the nozzle; however, the printing temperature was limited to 185°C as the binder decomposition onset temperature is 190°C based on STA analysis (Fig. 2a). In the case of Al-Kcmix, good flowability was achieved at 140°C which is slightly below the binder decomposition onset point.

The quality of the printed parts was assessed through optical microscopy of their cross-sections. As shown in Fig. 6a, the presence of pores and interlayer delamination was observed in samples, indicating

issues related to material flow and layer adhesion during printing. However, as illustrated in Fig. 6b, increasing the extrusion multiplier from 2.7 to 3.9 significantly reduced the gaps between the printed layers as more material was extruded. Similar findings have been reported by Hadian et al. for zirconia-based feedstocks [24]. This adjustment enhanced interlayer bonding and overall part density, suggesting that careful tuning of extrusion parameters like temperature and multiplier are crucial for improving the structural integrity of printed parts. All selected printing parameters for each feedstock have been summarized previously in section 2.2 (see Table 2).

According to the guidelines provided by the binder manufacturer (section 2.3), a two-step debinding including solvent and thermal debinding is required for the printed parts. Thermal debinding is usually carried out in air, as the organic components can react with oxygen, which facilitates the decomposition process. In the case of Al powder, however, oxidation occurs above 400°C in air (see Fig. 1b). Therefore, to maximize binder removal while minimizing powder oxidation, a wick debinding step in air up to a maximum temperature of 400°C was investigated. Final thermal debinding was performed under N₂ to prevent oxidation.

For the Al–EnCeram feedstock, water was used as the solvent in the solvent-thermal debinding approach, as required by this particular binder composition. Although potential oxidation of the Al powder was a concern, the process was carried out in accordance with the manufacturer's guidelines (Section 2.3), achieving a binder loss of ~ 52 wt.% after 48 h of immersion. The Al–Kcmix feedstock underwent solvent debinding in a high-purity acetone bath at 40°C for 24 h, removing approximately 67 wt.% of the binder, as reported in Fig. 7. Notably, no deformation or defects, such as cracking or blistering, were observed after this step. Subsequent thermal debinding in air at 350°C for 24 h was applied to both feedstocks, achieving a total binder removal of ≥ 96 wt.%.

Sintering of the debound samples was carried out at 635°C for 2 h. To evaluate the sintering performance, the final density of the as-sintered parts was measured and presented in Fig. 7. As expected, the results for the Al–EnCeram showed that solvent debinding in water led to a significantly reduced density after sintering, approximately 75%. SEM analysis of the sintered samples (Fig. 8a) highlights a thick, porous surface layer, consisting of individual grains not attached. In contrast, neck-formation between the particles was evident at the inner part, confirming densification. Based on this microstructure, it can be inferred that the aluminum powder in the outer layers acts as an oxygen getter for the powder in the inner layers [26]. When oxidation occurs in metal powders, a surface oxide layer forms which limits effective sintering [27]. In addition, as received Al powder was immersed in water for a duration equivalent to the solvent debinding process, then filtered, dried, and characterized. SEM analysis (Fig. 8c) revealed a rough layer on the powder surface, having a different morphology compared to the smooth as-received powder (Fig. 1a). Similar findings are reported in the literature [28, 29]. XRD results (Fig. 8d) further confirmed that aluminum reacted with water to form aluminum hydroxide (Al(OH)₃). Upon subsequent thermal debinding, this hydroxide transforms into aluminum oxide (Al₂O₃). The excessive growth of this passive oxide layer on the particle surface acts as a diffusion barrier, thereby hindering effective sintering [27].

For Al-Kcmix samples, solvent debinding in acetone followed by thermal debinding led to a sintered density of about 94% (Fig. 7). Figure 8b shows a better densification in the middle of the sample. However, the surface of the sintered Al-Kcmix sample could not be densified either. Therefore, using a commercial binder that can be partially debound in higher purity acetone does not improve surface densification, indicating the requirement of further optimization in the debinding process.

As an alternative approach, a single-step debinding route was evaluated, eliminating the solvent debinding stage and employing debinding solely through wicking embedment [30–35]. Wicking agents can be porous solid substrate plates or loose powders and granules. A liquid spontaneously flows from bigger into smaller pores due to capillary forces. This occurs due to attractive forces between the liquid and the solid surface of the pores, as well as the surface tension of the liquid.

The single-step wick debinding process was investigated for both feedstocks. Although the diffusion behavior of binder components through the printed parts differs between thermal and wick debinding, the thermal decomposition of the binder additives remains unaffected. To evaluate the feasibility of one-step wick debinding, kinetic analysis was performed on printed parts both with and without prior solvent debinding (Fig. 9a-d). For Al-EnCeram system, the sample without solvent debinding exhibited a lower activation energy than the solvent-debound counterpart (Fig. 9a). Examination of the conversion rate along the heating profile revealed a pronounced peak for the non-solvent-debound sample, indicating a rapid reaction event. This behavior suggests the occurrence of an exothermic process that releases a substantial amount of energy, thereby facilitating binder removal and reducing the apparent activation energy. The presence of this exothermic reaction is further confirmed by differential DSC measurements provided in the Supplementary Information (Fig S1), which show a distinct exothermic peak within the same temperature range. Consequently, the lower activation energy observed for the sample without prior solvent debinding can be attributed to internally generated heat during thermal decomposition (Fig. 9b).

A similar trend was observed for the Al-Kcmix system. After reaching a conversion of approximately 0.5, the activation energy of the solvent-debound specimen exceeded that of the sample without prior solvent debinding (Fig. 9c). Careful examination of the conversion rate versus time curve revealed a pronounced peak at this stage, again associated with an exothermic reaction during debinding. Correspondingly, DSC results in Supplementary Information (Fig S2) also display an exothermic signal in this temperature interval, supporting the kinetic analysis. The heat released accelerates binder decomposition, resulting in a lower apparent activation energy for the non-solvent-debound sample. Nevertheless, for both binder compositions, the maximum conversion rate remained below $0.8\% \text{ min}^{-1}$ during heating (Fig. 9b and d). Based on our findings, conversion rates below this threshold do not impose detrimental stress on the printed structure, indicating mild and gradual thermal degradation. It should be noted that the actual binder removal behavior may differ from the kinetic predictions, as capillary effects present during wick debinding are not captured in thermal analysis. Therefore, slightly lower conversion rates are expected in practice compared with those shown in Fig. 9b and d.

After debinding, the wick-debound samples exhibited no deformation or defects, such as cracking or blistering. As shown in Fig. 9e, for both Al–EnCeram and Al–Kcmix at 350°C, a binder loss exceeding 97 wt.% was recorded. Interestingly, the amount of binder removed at 400°C was lower reaching 92 wt.% for Al–EnCeram and 96 wt.% for Al–Kcmix. Considering that the thermal debinding process maintained the samples at certain temperatures (350 and 400°C) for 24 h, TG analyses for aluminum powder were performed for 24 h at constant temperatures 350 and 400°C, respectively. As shown in Fig. 10, a mass increase of approximately 0.25% was observed after 24 h of 400°C heat treatment, attributed to oxidation of the particle surface, inhibiting diffusion-driven mechanisms of metal sintering. At 350°C, no mass gain was identified, meaning no oxidation at the surface of the aluminum particles occurred.

After single-step wick debinding, further heat treatment, including sintering was carried out under N₂ atmosphere. Table 3 shows the sintering conditions and the achieved relative density after sintering of the Al–EnCeram and Al–Kcmix samples. Two different dwell times, namely 2 and 10 h, were investigated. The sintering results showed that the Kcmix-350-2 printed disc, using the alternative wick debinding approach, achieved a higher relative density of 96.7%. The densities obtained in this study for both binder systems exceed most values reported in the literature for additively manufactured aluminum feedstocks. Maolne [15], reported relatively low densities in the range of 47–70 %, whereas Ding et al. [36], achieved relative sintered densities of 93–96 %. Looking on the cross-section of this sample presented in Fig. 11a, a uniform sintered microstructure is visible with no sign of porous skin formation. EnCeram-350-2 sample reached a lower relative density of 93.6 % compared to Kcmix-350-2. Microstructural analysis presented in Fig. 11b, shows higher porosity between the grains in this sample which caused a lower density. This is likely due to short dwell time, or insufficient sintering temperature as described by J. Liu et al. [37]. In both samples, a secondary phase is observed at the grain boundaries. According to Z. Liu et al. [3], this phase is AlN. This phase can be formed under carefully controlled atmospheres, facilitated by the presence of magnesium acting as a getter in the Al alloy composition. Its occurrence along grain boundaries is significant, as it suppresses grain growth during the sintering process. The larger voids in both samples are related to the sample preparation and were caused during the polishing step.

Table 3

3D printed sample designation under different wick debinding and sintering dwell time at 635°C

Sample	Binder system	Wick debinding temperature (°C)	Sintering time (h)	Relative density (%)
EnCeram-350-2	EnCeram	350	2	93.6
EnCeram-350-10	EnCeram	350	10	96.8
EnCeram-400-2	EnCeram	400	2	81.2
Kcmix-350-2	Kcmix3.3	350	2	96.7
Kcmix-350-10	Kcmix3.3	350	10	97.9
Kcmix-400-2	Kcmix3.3	400	2	77.4

As expected by the TGA analysis shown in Fig. 11, increasing the wick debinding temperature 350 to 400°C, a lower relative sinter density was achieved. Therefore, the lower relative sintering density can be explained by surface oxidation of Al powders.

To further improve the sintered density, the effect of increasing the holding time from 2 to 10 h was investigated. The relative density of the Kcmix-350-10 samples increased to 97.9%, while the EnCeram-350-10 samples reached a relative density of 96.8%. The cross-sections of both samples (Fig. 12a, b) reveal homogeneous densification throughout the specimens, with no significant differences between the center and edge regions. In addition, expanded grains that have lost their initial spherical shape could be observed. The positive influence of the extended sintering time on the final density is in good agreement with previous results reported by Acar et al. [38].

4. Conclusion

This study highlights the potential and challenges of extrusion-based 3D printing for Al6061 alloys, emphasizing the critical role of processing parameters, binder characteristics, and post-processing in achieving defect-free, high-density aluminum parts. The binder removal process significantly influences the later sintering process. A step conventional debinding process, including solvent debinding, leads to surface oxidation and poor densification. Microstructural analyses revealed regions of incomplete densification and the presence of pores, as well as the formation of necks and well-defined grain boundaries during sintering. Single-step wick debinding enabled gradual binder removal, a homogeneous sintered density in the center and the edge of the samples, a reduction in porosity and a high densification of 98% after sintering. Wick debinding at 350°C and extended sintering times improved densification. These findings demonstrate that a carefully controlled debinding process will be a good solution to use commercial thermoplastic binder systems for high-density Al6061 components.

Overall, extrusion-based 3D printing of aluminum 6061 demonstrates promise as a cost-effective route for fabricating complex geometries. The results of this study are not limited to 3D printed objects. The feedstocks with commercial binder can also be shaped by warm pressing, extrusion or injection molding.

Declarations

Author Contribution

E.M.: Conceptualization, Methodology, Validation, Formal Analysis, Writing – Original Draft, Writing – Review & Editing, Visualization, Supervision.S.L.: Methodology, Software, Validation, Formal Analysis, Investigation, Data Curation.M.K.: Supervision.A.H.: Conceptualization, Writing – Review & Editing, Supervision.F.C.: Conceptualization, Methodology, Resources, Writing – Review & Editing, Supervision, Project Administration, Funding Acquisition.All authors reviewed the manuscript.

The authors declare that they have no competing interests.

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Figures

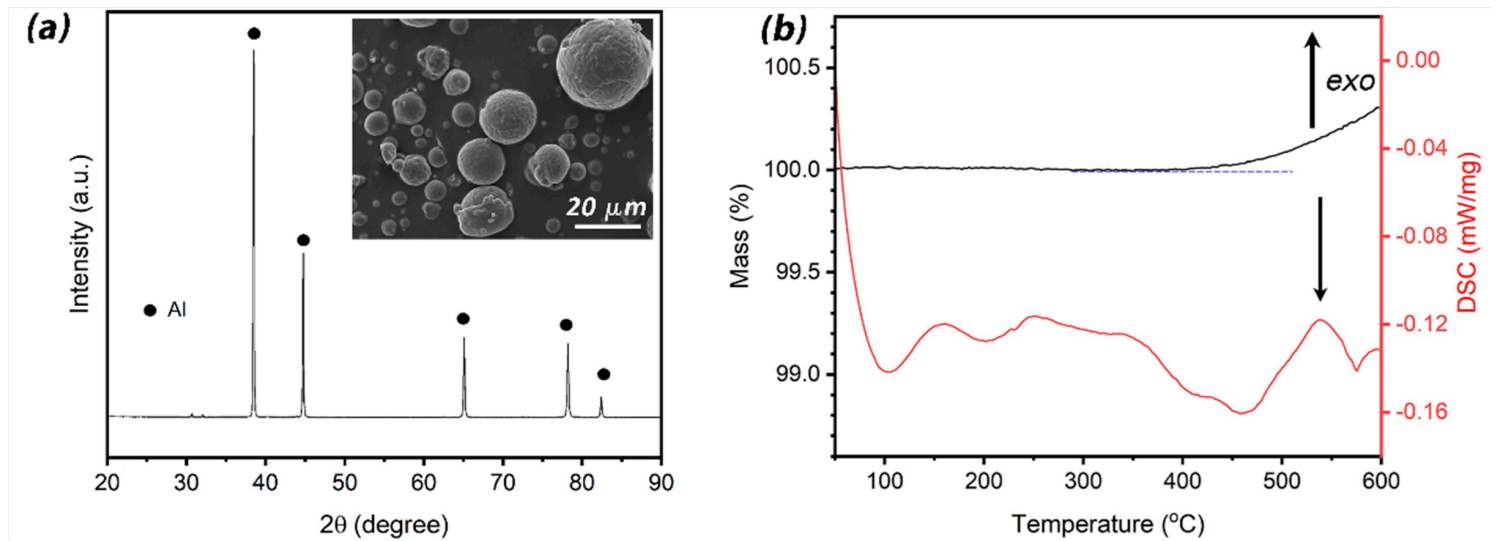


Figure 1

As received aluminum powder a) XRD analysis with an inset of SEM image revealing the spherical morphology and b) TG/DSC under a synthetic air flow.

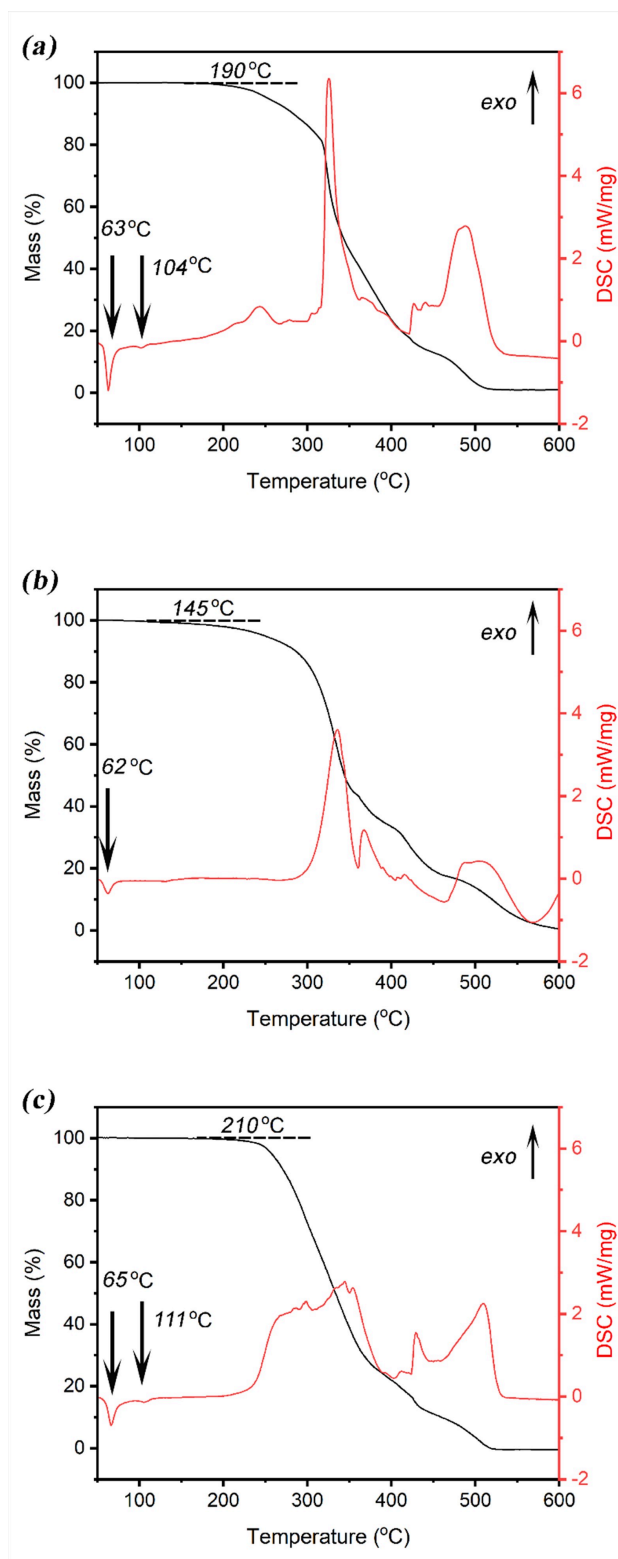


Figure 2

TG–DSC analysis of (a) EnCeram, (b) Kcmix, and (c) EmbeCC, conducted up to 600 °C under a synthetic air flow.

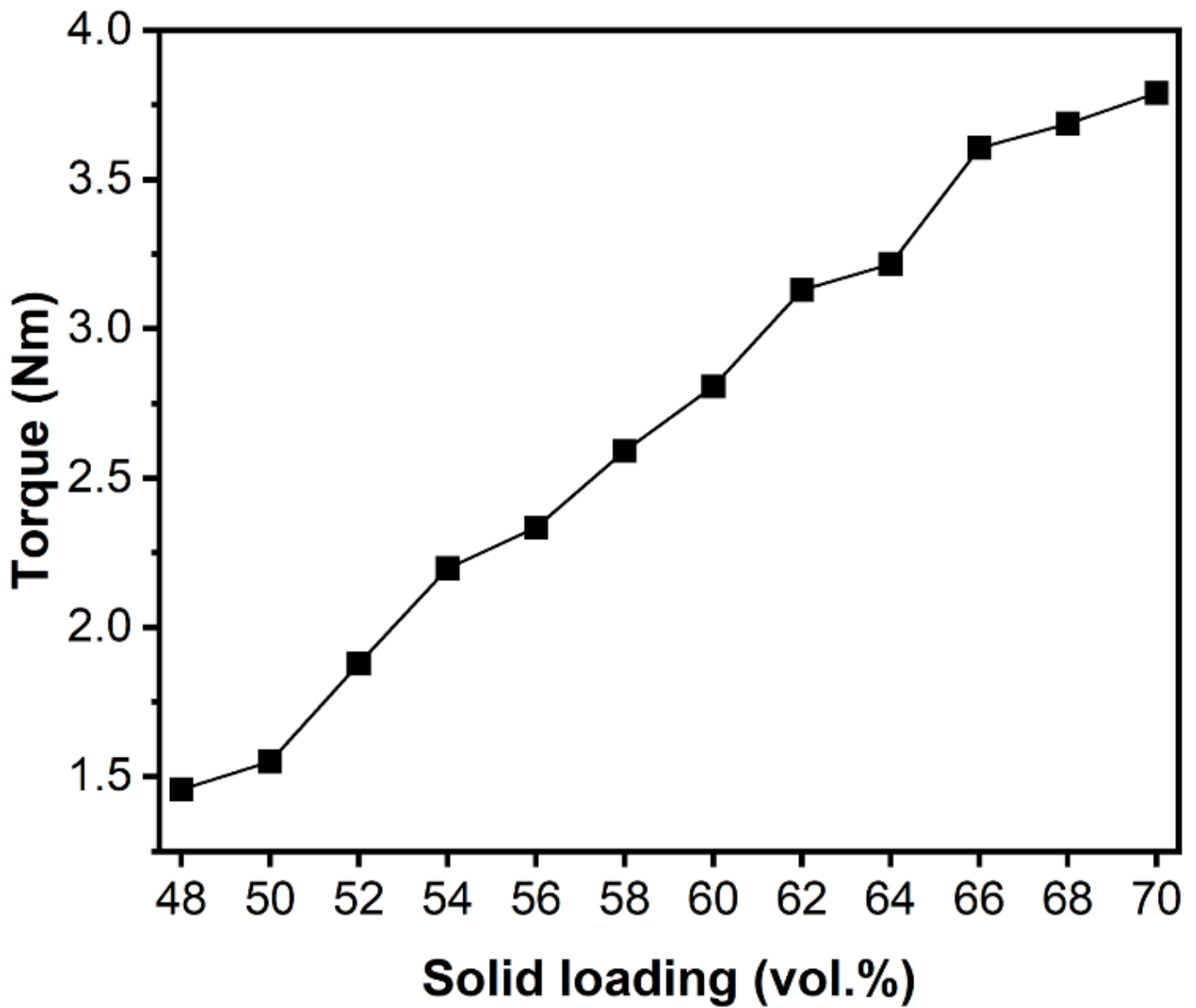


Figure 3

Variation of mixing torque with increasing solid loading of Al powder during feedstock preparation using the EnCeram binder composition.

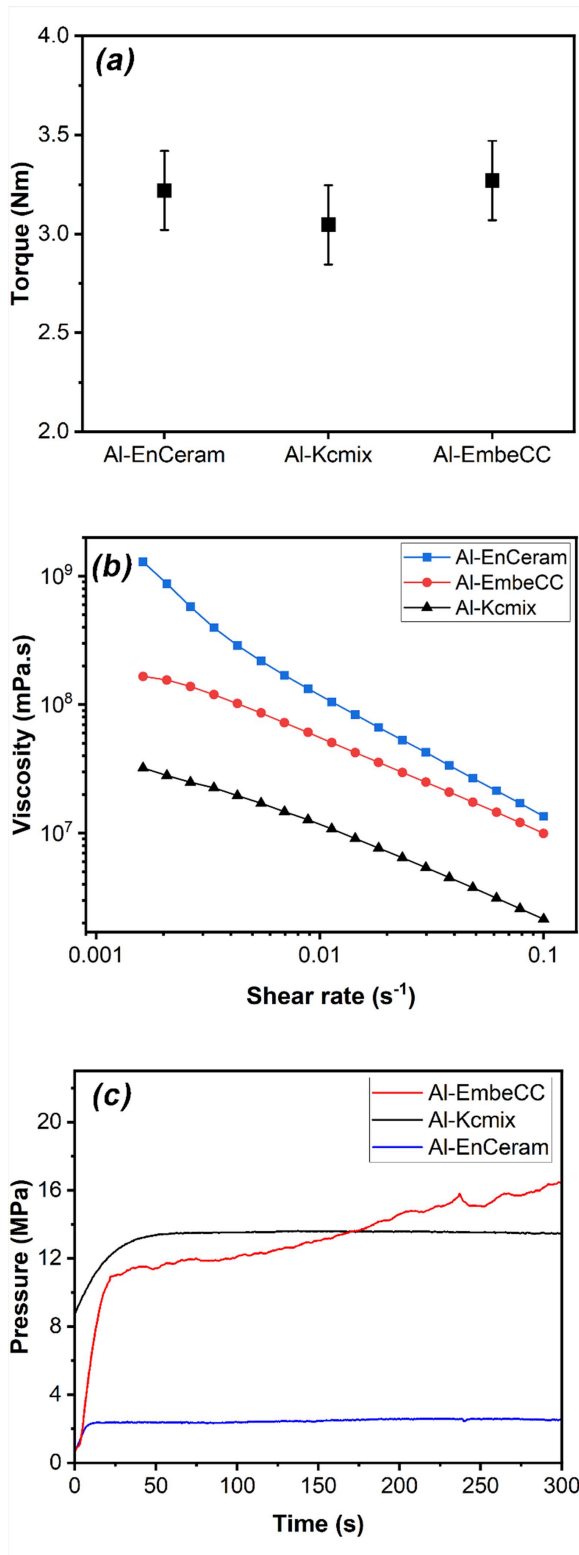


Figure 4

(a) Average torque at the equilibrium of the mixing process at $\sim 100^\circ\text{C}$ for each feedstock composition. (b) Viscosities of prepared feedstock at two different temperatures 180°C for Al-EnCeram and Al-EmbeCC and 130°C for Al-Kcmix, in the shear rate range of 0.001 to 1 S^{-1} and (c) Extrusion pressure for feedstocks containing Kcmix and EmbeCC as binder component at 140°C and EnCeram binder at 160°C .

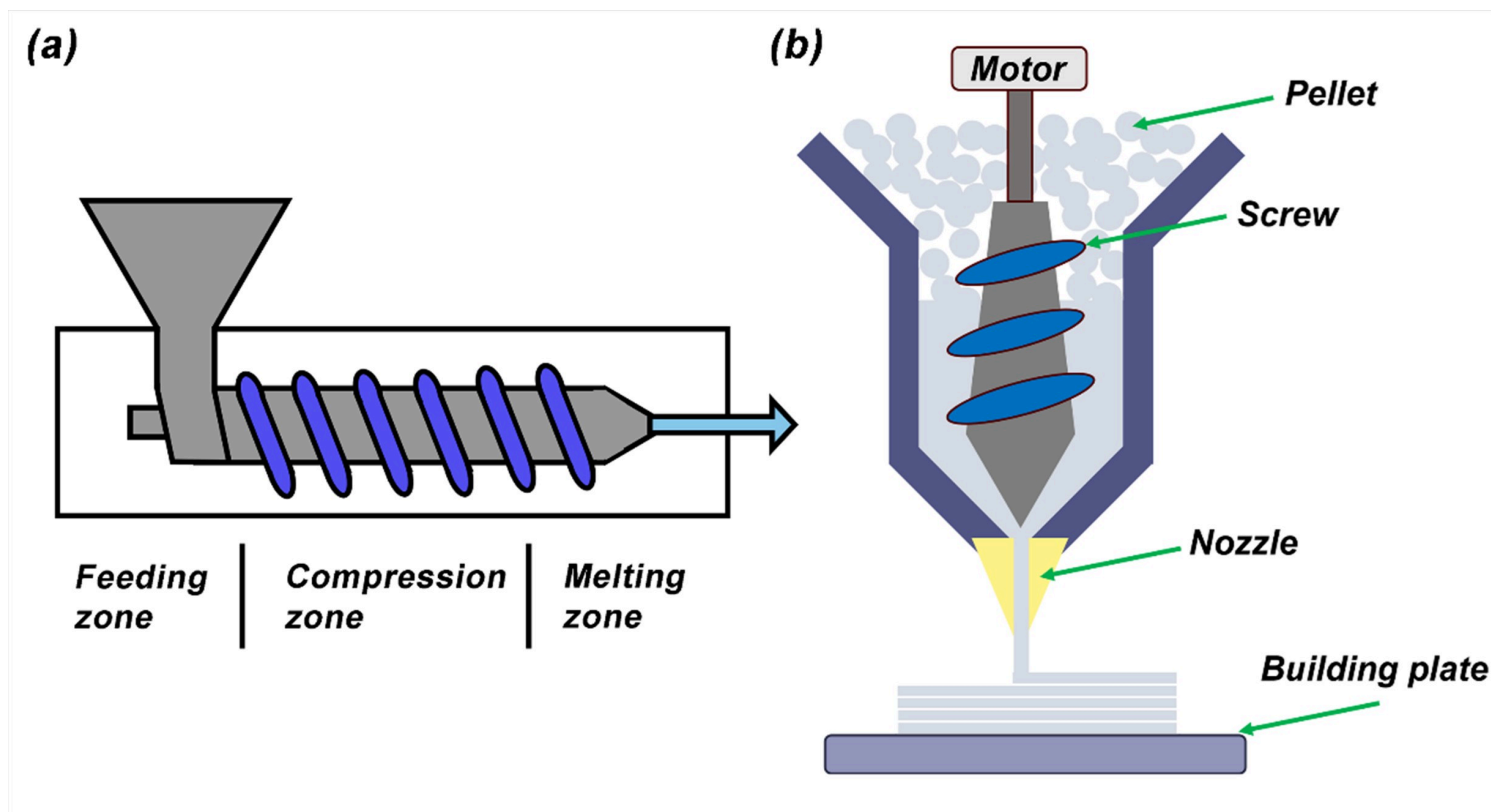


Figure 5

Schematic representation of a pellet-screw extruder: a) feeding, compression, and melting zones; b) integration within a 3D printing system.

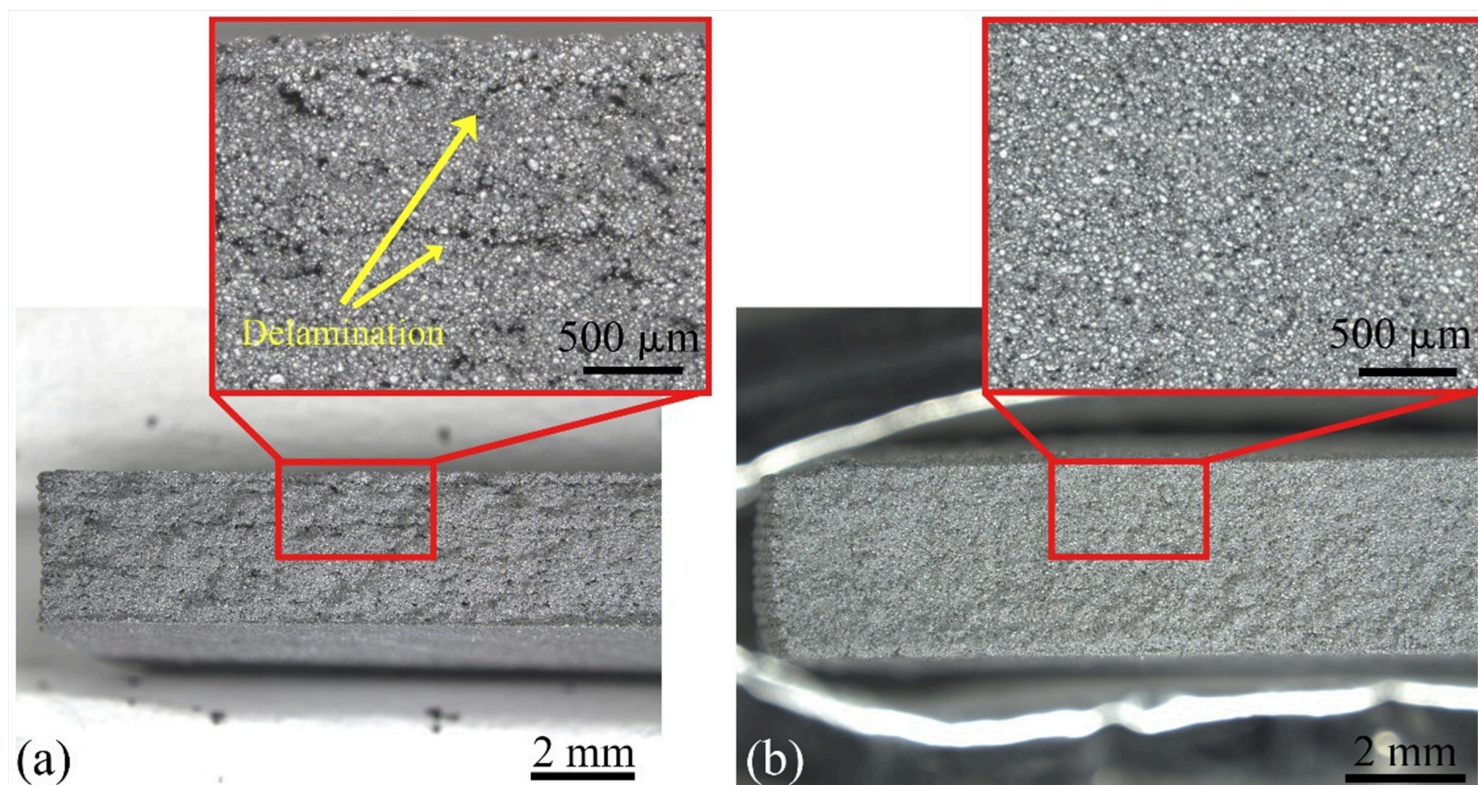


Figure 6

Cross-section of an as-printed parts using Al-EnCeram feedstock: a) delamination and pores caused by low extrusion multiplier (2.7), b) no print-defects using higher extrusion multiplier (3.9).

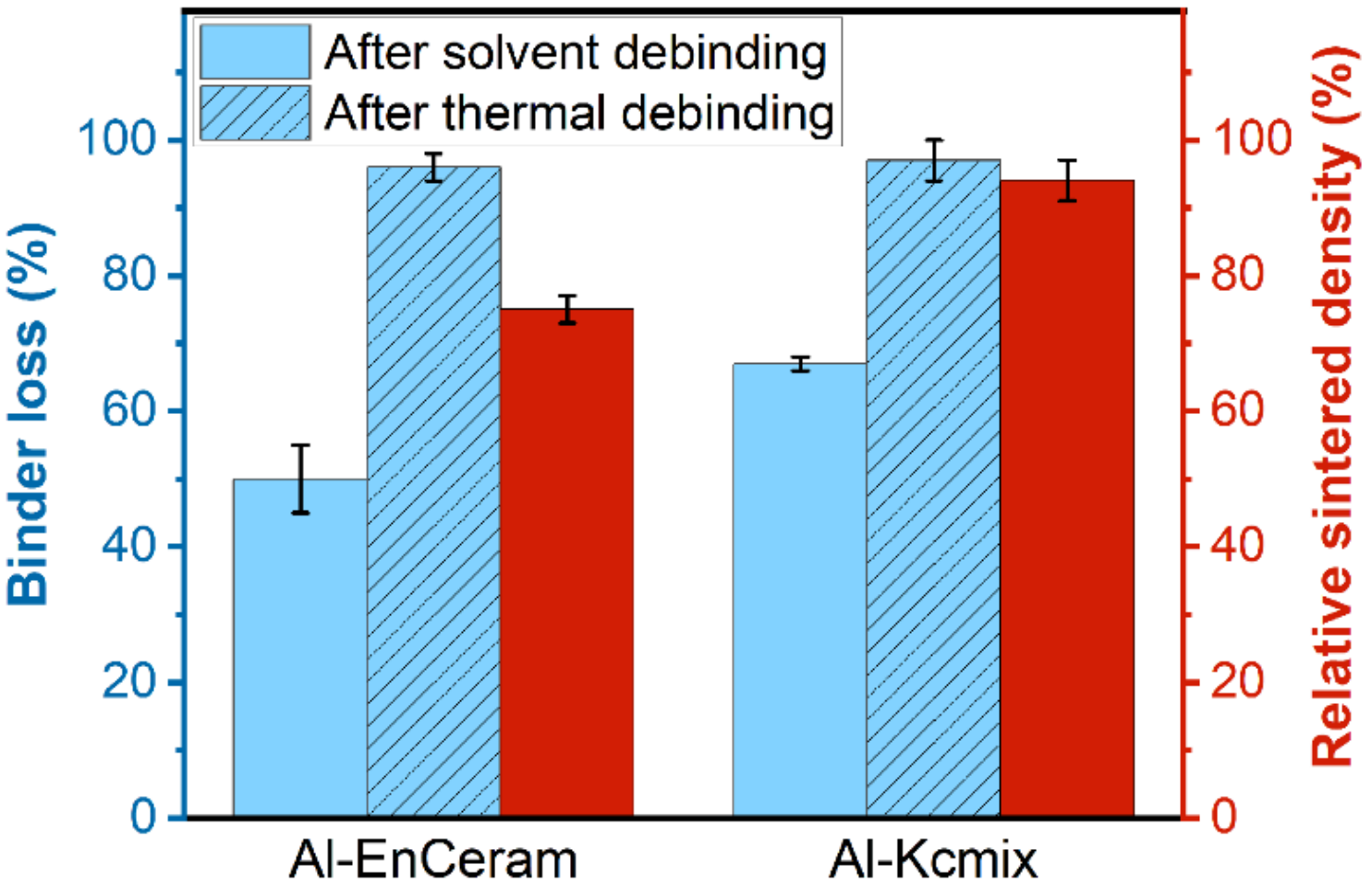


Figure 7

Binder loss after each step of debinding and relative density after sintering at 635 °C for 2 h under N₂ flow.

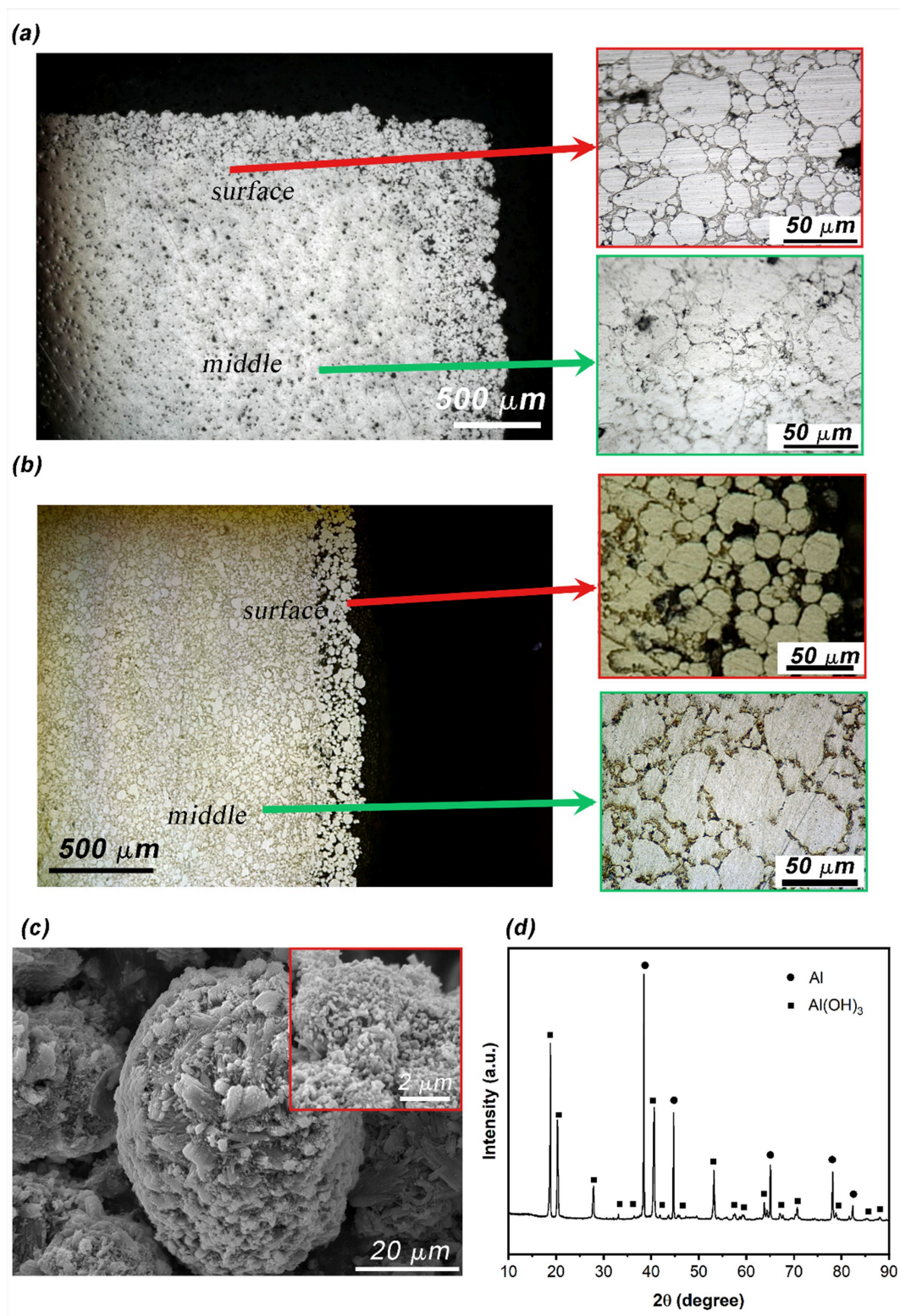


Figure 8

Optical microscopic images of the sintered (a) Al-EnCeram, (b) Al-Kcmix printed part. (c) SEM and (d) XRD analysis of Al6061 powder after immersing in water.

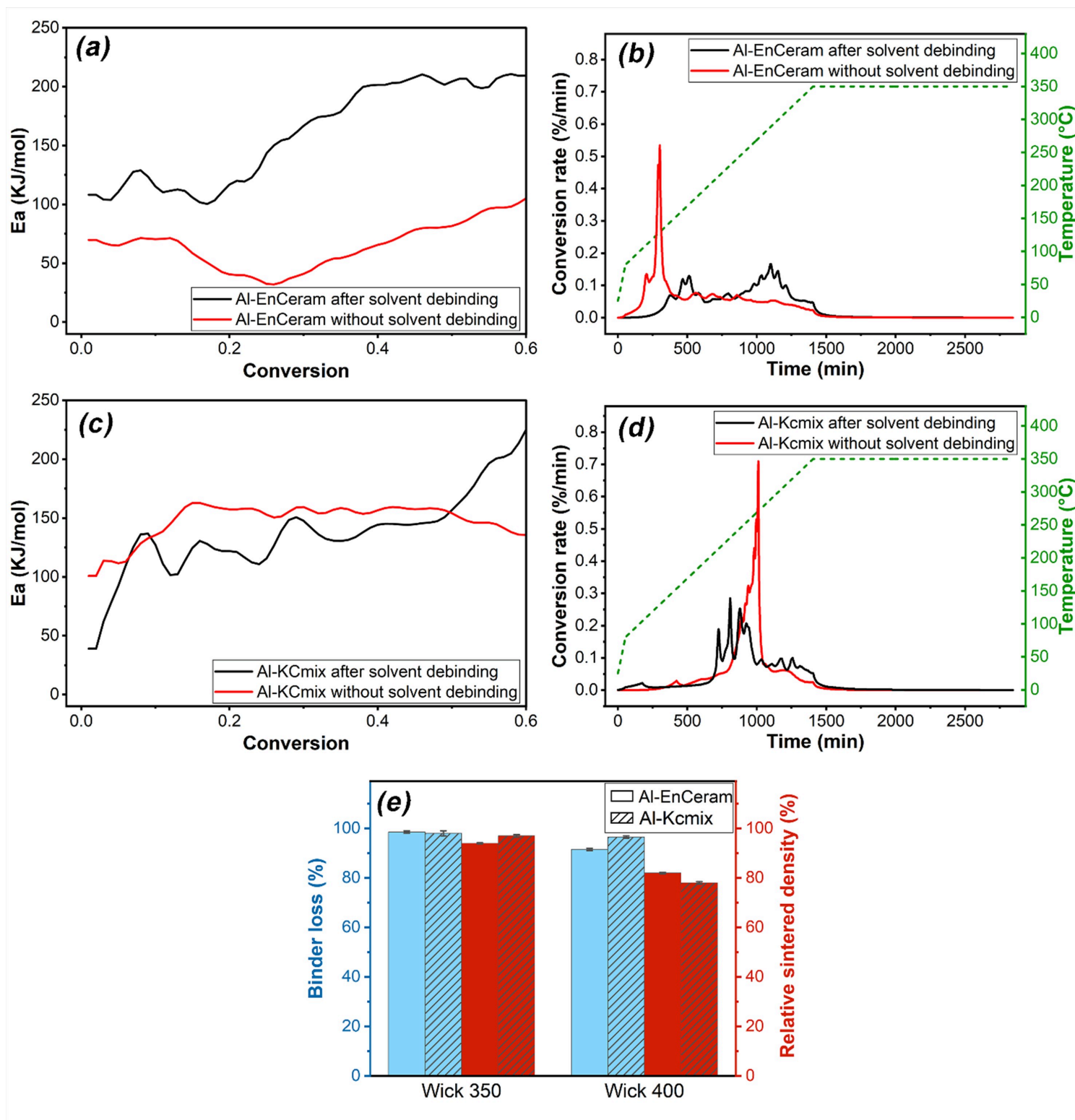


Figure 9

Activation energy (a,c) and conversion rate (b,d) from kinetic analysis of Al-EnCeram (a,b) and Al-Kcmix (c,d) printed parts before and after solvent debinding. (e) Binder loss after wick debinding at 350 and 400 $^{\circ}$ C and relative density after sintering at 635 $^{\circ}$ C for 2 h under N_2 .

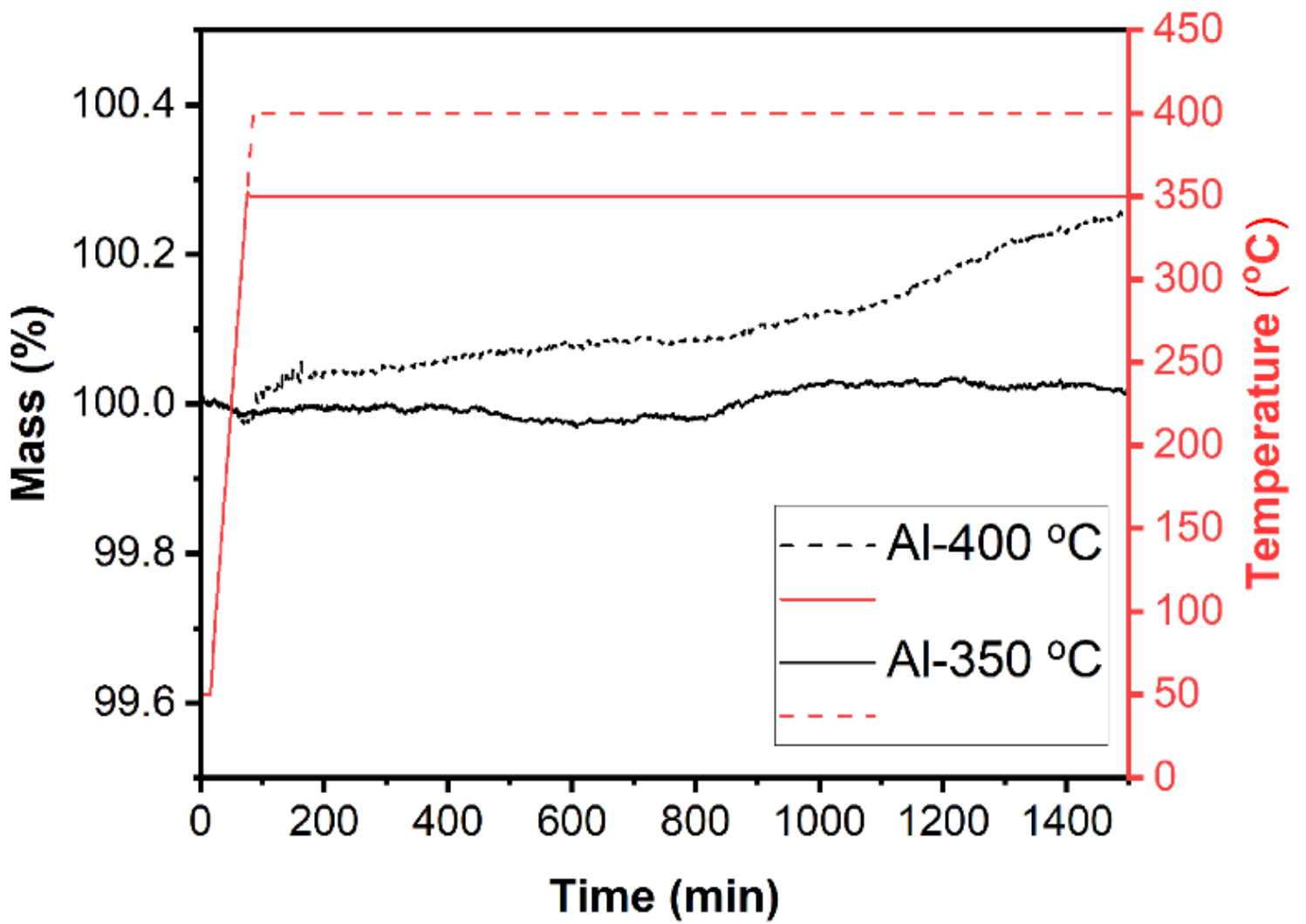


Figure 10

TG analysis of the Al powder at 350°C and 400°C after heat treatment for 24 h under a synthetic air flow.

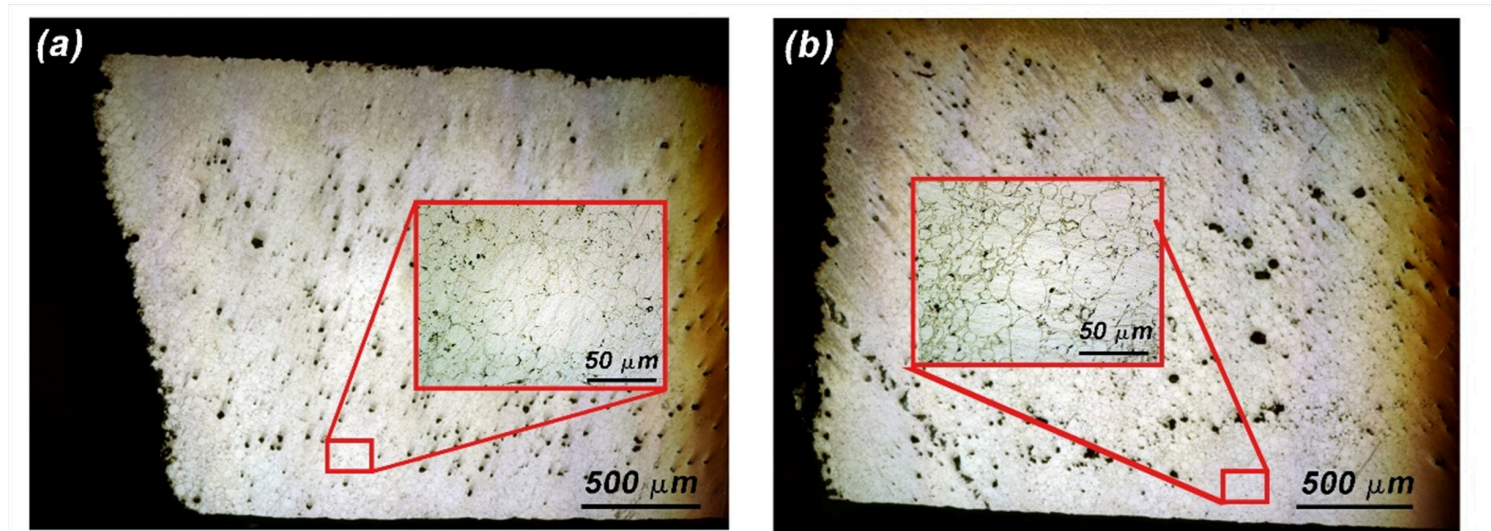


Figure 11

Optical microscopic images of the (a) EnCeram-350-2 and (b) Kcmix-350-2 samples.

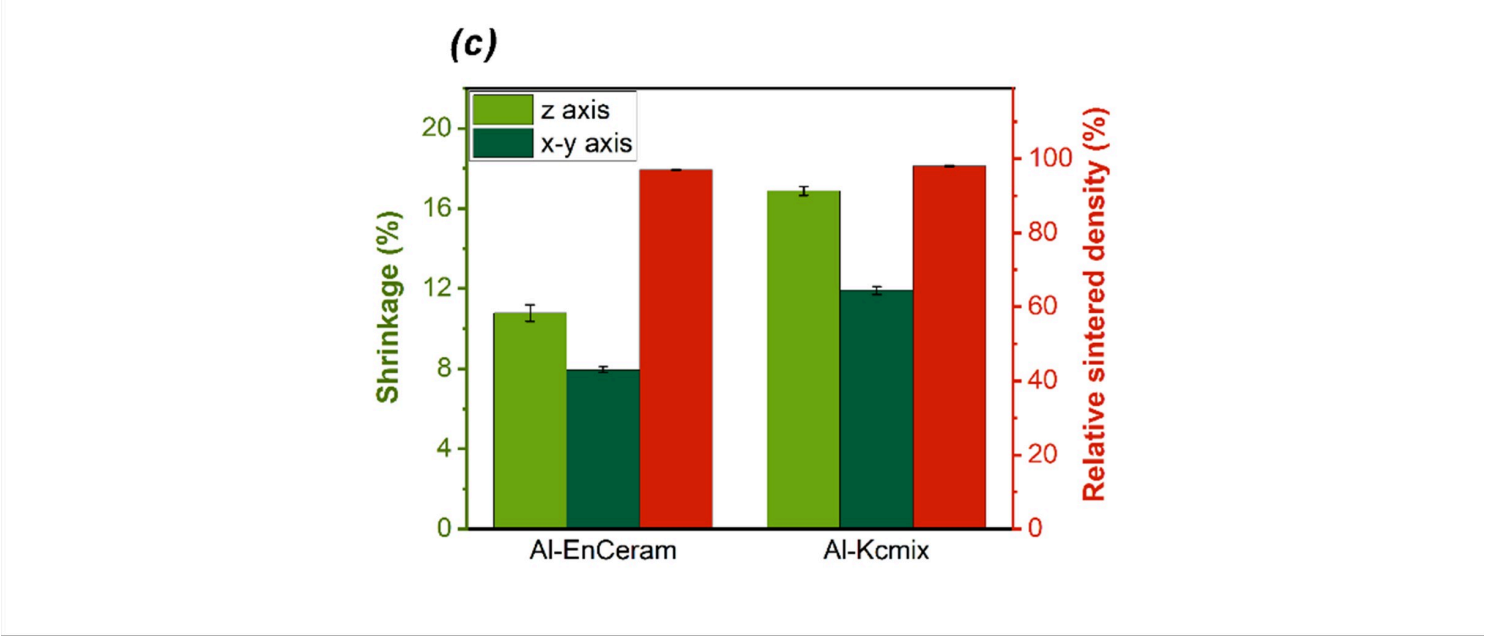
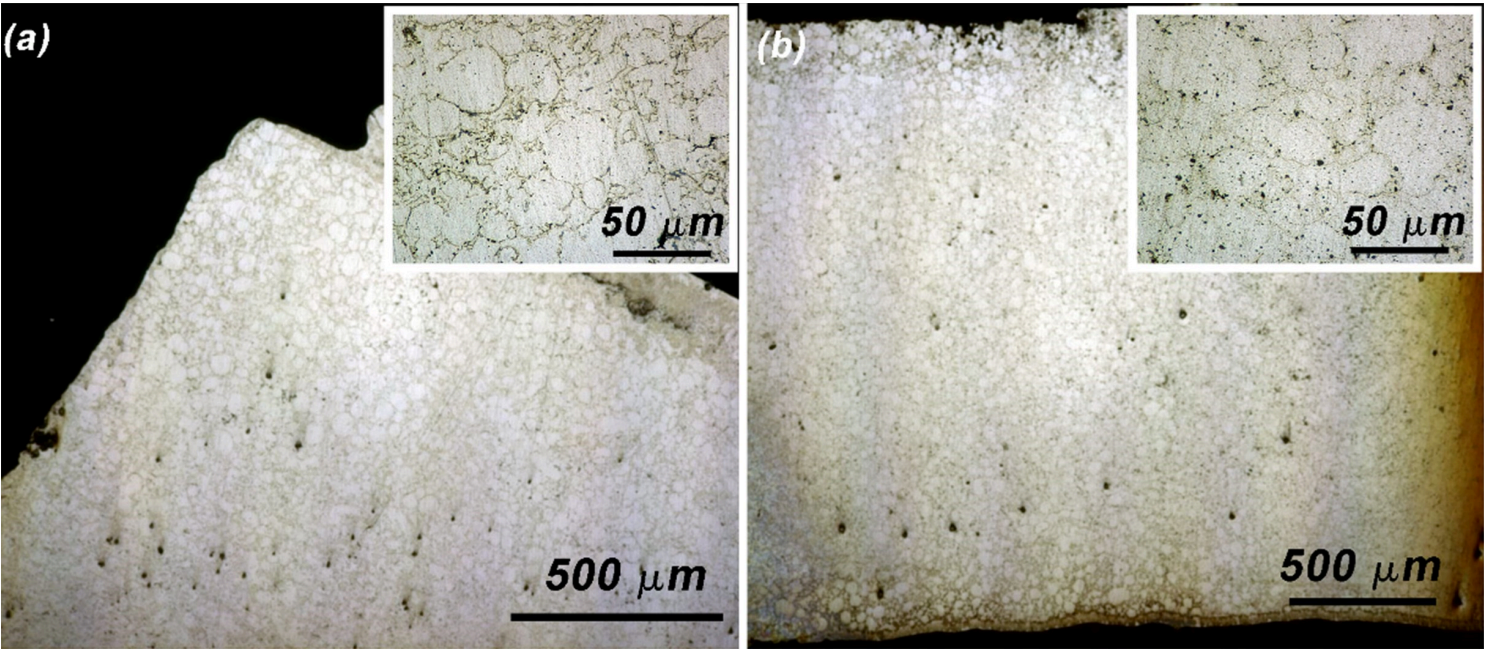


Figure 12

Optical microscopic images of (a) EnCeram-350-10, (b) Kcmix-350-10 and corresponding measured (c) shrinkage and relative density of the samples sintered at 635°C for 10 h.

Supplementary Files

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