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RESEARCH

A systematic approach to evaluate jetability of high-viscosity resins for 3D inkjet printing applications

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Abstract

Recent advances in piezo inkjet printing technology have enabled the use of high-viscosity materials previously unsuitable for inkjet applications. This technology facilitates the printing of vat photopolymerization resins, traditionally used to produce single-material components with superior properties compared to those achieved with conventional UV inks in material jetting. The ability to print these resins with piezo inkjet technology opens new fields of application for producing multimaterial functional parts. To fully exploit this potential, it is essential to evaluate the compatibility of these resins with the new technology. This study systematically evaluates the jetability, printability, and performance of high-viscosity materials to optimize the printing process and provides a detailed understanding of the factors influencing the jetability of high-viscosity resins and develops guidelines for optimizing their use in 3D inkjet printing applications.

Keywords Inkjet · Rheology · Resin · Ink · Material jetting

1 Introduction and background

Additive manufacturing (AM) exceeded its limitation to prototyping and has found its way into manufacturing more recently. Medical applications are an example of AM being utilized to manufacture individualized products for patients' needs. Nearly all seven AM techniques classified in ISO/ASTM 52900 by their functional principles are applied in the medical field [1]. Powder bed fusion (PBF), material extrusion (MEX), and vat photopolymerization (VPP) are all commonly used in various medical applications such as implants and prostheses, medical models, or tools. Direct energy disposition (DED) and sheet lamination (SHL) on the other hand are rarely applied. Material jetting (MJT) is used for medical models due to its high accuracy of results. However, the range of materials that can be used is highly limited, which is the reason for MJT not being suitable for most functional prints, such as implants and prostheses [2].

Generally, with most AM techniques they are limited through the materials that can be utilized. Especially limiting is the number of building materials employed in a single process. Except for extrusion and jetting all of them are only able to work with single-material approaches. However complex functional prints need an exact deposition of multiple materials to be able to create 3D structures with finely tuned properties. A possible use would for example be the manufacturing of realistic dentures with exact color and property replication.

Dentures are currently produced by VPP with a single material throughout the prosthesis. To achieve a photorealistic and functional denture the VPP resins could be deposited with the accuracy and multimaterial approach of inkjet printing. However, especially inkjet-based material jetting processes are heavily limited by the material properties that can be worked with. Piezo inkjet printheads operate based on the inverse piezoelectric effect, where an electrical impulse causes mechanical deformation in

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an ink chamber via a piezo actuator, resulting in the ejection of ink droplets. The size and velocity of the droplets are influenced by the printhead design, material rheology, and the electrical signal. Inkjet printheads developed over the last twenty years require a precisely adjusted viscosity range of 10–20 mPa·s, with typical tolerances of around ± 2.5 mPa·s. The viscosity of a fluid changes with temperature, e.g., a 5 °C change can increase or decrease the viscosity by up to 20 mPa·s or more, depending on the ink. This property can help to adjust the viscosity of the ink to a printable range, but it also presents a challenge as temperature fluctuations can lead to unstable jetting performance. In addition, exposing UV inks to high temperatures to achieve a jettable viscosity can compromise ink stability. The inks are typically tuned to exhibit Newtonian behavior [3]. Functional materials on the other hand, usually exhibit a very different rheological behavior. As an example, VPP resins used for the manufacturing of dental prostheses have a viscosity of roughly 1000 mPa·s at room temperature. These resins are also highly filled with ceramic particles to achieve the desired toughness in the final print of a dental prosthesis. These materials cannot be printed with conventional piezo-based inkjet printheads. However, recent developments by manufacturers such as Seiko and Xaar have enabled printing with higher viscosities, around 20–30 mPa·s, and in special cases, even up to 100 mPa·s with a precisely tuned waveform and optimized fluid handling at jetting temperatures [4] [5]. In addition, the particle size remains a limiting factor for all of these printheads.

Recently a novel technology was presented by Quantica GmbH in the form of the NovoJet™, which is a printhead that can jet fluids up to 4500 mPa·s (measured at room temperature), which equals a viscosity of max. 250 mPa·s at jetting temperature and has a nozzle diameter of 50 µm which opens up the possibility to jet fluids with higher viscosity and particle load [6]. Typical inkjet materials for conventional printheads are characterized mostly by their viscosity and surface tension to determine the printability. The most used characterization is the Ohnesorge number to predict whether a fluid is jetable. Equation (1) shows the calculation of the Ohnesorge number using the viscosity η, the density ρ, and the surface tension σ of the fluid, as well as the diameter d of the fluid cylinder or the nozzle, respectively.

$$\mathrm{Oh} = \frac{\eta}{\sqrt{\rho \bullet d \bullet \sigma}} \tag{1}$$

A fluid is classified as jetable if the Ohnesorge number is $0.1 < \mathrm{Oh} < 1$. These calculations however are only applicable to Newtonian fluids [7]. Materials for functional prints on the other hand rarely exhibit Newtonian behavior due to, for example, high particle load or longer polymer chains causing viscoelastic properties.

Increased viscoelasticity introduces significant challenges in the detachment dynamics at the nozzle plate, leading to an extended droplet ligament, with the pinch-off to form a discrete droplet occurring at a delayed stage. Consequently, as elasticity increases, the predictability and control of droplet detachment become increasingly complex, making it more difficult to achieve a well-defined and consistent droplet formation as shown in Fig. 1. This presents a particular challenge in the processing of VPP resins, as longer polymer chain length are typically used to improve properties such as mechanical strength or temperature resistance [9].

The dynamics behind jetting such challenging materials are yet to be explored. This work aims to present a means to assess the jetability of materials through the connection of rheology and waveform parameters.

In previous work, we were able to show that standard VPP resins usually consist of a polymer base that shows Newtonian behavior and mostly gets its viscoelastic properties from the particles (fillers and pigments) inside. To perform tests at a suitable temperature and to provide accessibility, this is done with test inks to provide a simple approach for this method that can be replicated without the need for hazardous and expensive UV resins or inks.

2 Materials and methods

Two VPP resins, material A and material B, were chosen for rheological characterization. For both resins, there was also a version prepared by the manufacturer that did not contain any pigments or fillers, base A and base B, to understand the origin of the viscoelastic properties of the materials. For the first jetting trials, PEG 400 as a test ink was chosen

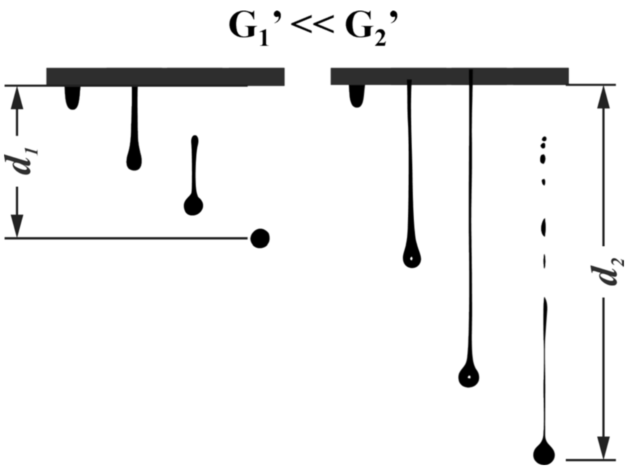

Fig. 1 Lower storage modulus (G_1') versus higher storage modulus (G_2') drop formation (Figure according to [8])

according to the results from the rheological characterization of the VPP resins.

To evaluate the rheology of the VPP resins first the shear viscosity η was measured using an Anton Paar MCR 302 rheometer. The viscosity was measured at different temperatures from 25 to 70 °C in a shear rate sweep from 100 to 10,000 1/s. Measurements at higher shear rates were not possible with the Anton Paar MCR 302 setup, because the high particle load in the dental resins caused agglomerations and subsequently scratches on the measuring stamp at shear rates above 10,000 1/s.

The next step in the evaluation of jetability is capturing the rheological properties of the materials and finding possible parameters for jetting. Since measurements at higher shear rates were not possible with a mechanical rheometer, a different setup is necessary to capture rheological properties at timescales relevant to the inkjet printing process. This is possible with high-frequency rheology, for example with a piezo-axial vibrator. One option for this is the measurement with the TriPAV rheometer (TJ-PV 1009n) by TriJet Limited. The device uses the piezo-axial vibrator (PAV) to actuate the sample and detect the response equally. Compared to conventional mechanical rheometers it has the advantage of being able to reach higher frequencies of up to 10 kHz. It was used to determine the complex rheological properties (storage modulus G’, loss modulus G’’, and the complex viscosity Eta*) in a frequency sweep from 10 to 10,000 Hz.

In addition, the piezo rheometer by TriJet Limited is able to perform a piezo actuation called “printhead mode” with the TriOSC setup (TJ-OS 3001 h). This measurement is employed to observe the oscillation and dampening response of the material similar to the actual actuation process in a piezo printhead. Here a temperature sweep from 40 to 70 °C for the dental resins and a sweep from 25 to 35 °C for the test ink is performed with an actuation of 2 V and a gap size of 200 µm. This gap size is chosen since it resembles the geometry of the Quantica NovoJet™ printhead the most.

3 Results and discussion

The first step in evaluating the jetability of high-viscosity fluids was developing a systematic approach to characterize those materials and then finding suitable test fluids to replace the materials in initial jetting trials. This is done because waveform development is a time-consuming and expensive process itself. On top of that functional materials are also costly. Test fluids should be inexpensive and less hazardous materials which also pose less risk of damaging a printhead or the dropwatching equipment.

In Fig. 2 the workflow that was systematically developed is shown schematically. The first step is the choice of materials. Especially for the application in the medical field the process of material testing and validation is lengthy and costly. Therefore, already established VP resins that are commercially available are promising materials if they can be jetted in a multimaterial process with little to no modification. In this work, two VPP resins from two different manufacturers were chosen, as well as the polymer base for these respective resins to evaluate the influence of the particle load. The next step is the rheological characterization of the chosen materials. These two blocks set the requirements for the test fluid set as shown in Fig. 2.

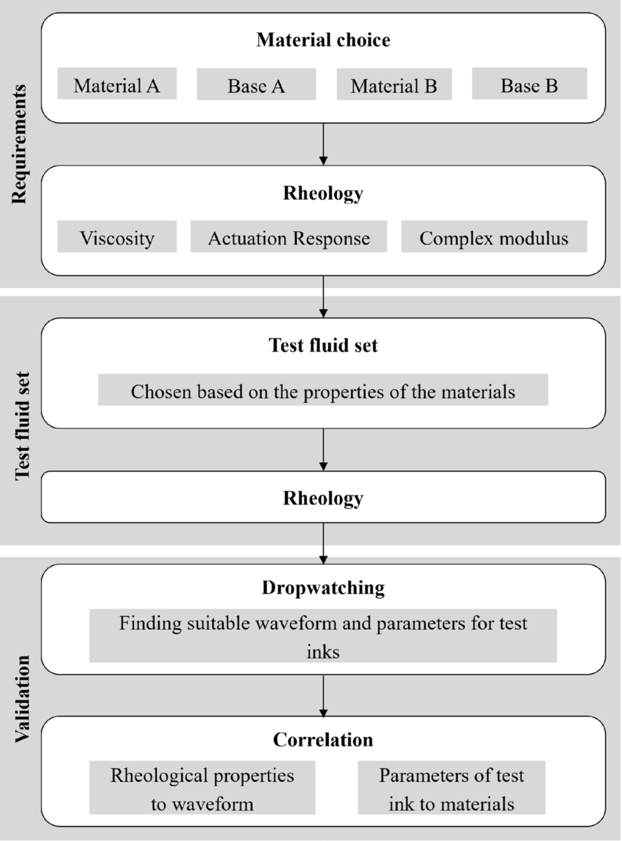

Fig. 2 Schematic workflow for the systematic characterization of materials for jetting

The rheological characterization of the commercially available VPP resins showed that they are shear thinning and have a viscosity of 1200–1500 mPa•s at room temperature at a low shear rate. The measurements of storage and loss modulus suggest viscoelastic behavior.

The rheological characterization of the base A and B showed that the viscoelastic properties must stem from the particles in the VPP resins as the base is entirely Newtonian. The viscosity of all four materials over a shear rate sweep is shown in Fig. 3 at 70 °C. This temperature is chosen since the viscosity of the materials is in a suitable range for the Quantica NovoJet™ printhead at this temperature. The material A and B have a viscosity of 60–70 mPa•s at 70 °C and a

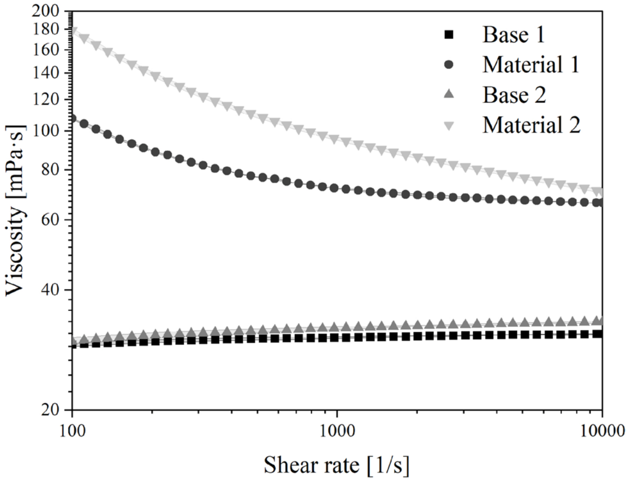

Fig. 3 Shear rate sweep of Material A and B, Base A and B from 100 to 10,000 1/s at 70 °C

shear rate of 10,000 1/s. The base A and B have a viscosity of 30 mPa•s, which is significantly lower than the viscosity of material A and B caused by the lack of fillers and pigments in the base. All four materials are in the jetable range of 1–250 mPa•s [10] at this temperature.

The Newtonian behavior of the base is also supported by the results of the measurement of the complex rheological properties with the TriPAV rheometer. Compared to a conventional mechanical rheometer this piezo-driven device can measure up to 10,000 Hz. This better represents the timeframe of the droplet formation in inkjet printing. An exemplary measurement of Base 1 and Material 1 is shown in Fig. 4. This shows a much higher storage modulus (G') for the Material 1 that has particles, compared to the low G' of the Base 1. The results for Base 2 and Material 2 are the same and therefore not separately displayed.

The other rheological measurement that is relevant to determine possible jetting parameters is the printheadmode measurement of the materials and bases with the TriOSC system. This measurement actuates the sample and then detects the reaction of the sample to said actuation. This is supposed to reproduce the movement in the printhead as well as the subsequent dampening. An example of a dampening curve captured with this measurement is shown in Fig. 5 for the Base 1 at 60 °C.

There is a pronounced first peak which is subsequently labeled as the peak amplitude. The height of this first peak is an indicator for the sample's initial response to a piezo actuation. The higher the peak the more likely the material is going to be jetted, as a higher peak indicates better energy transfer or absorption into the fluid. This response signal is captured over a temperature sweep and the peak amplitude is calculated. When plotting the peak amplitude over the temperature it can be determined from which temperature on a material is most likely to be jetted. This is shown for the two materials and their respective bases in Fig. 6 for a temperature range from 40 to 70 °C. Base A has a plateau of the peak amplitude starting from 50 °C. For Base B it is slightly higher with 55 °C. For the materials A and B the onset of the peak-amplitude plateau is at around 60 °C. This suggests that a droplet ejection is most likely from this temperature on according to the TriOSC measurement.

After the requirements have been set through the rheological characterization of the materials a test fluid set is determined in step 3, according to Fig. 2. This set is supposed to consist of a Newtonian fluid, a polymer solution with viscoelastic properties, and finally a particle loaded fluid. This is supposed to give a systematic approach to the

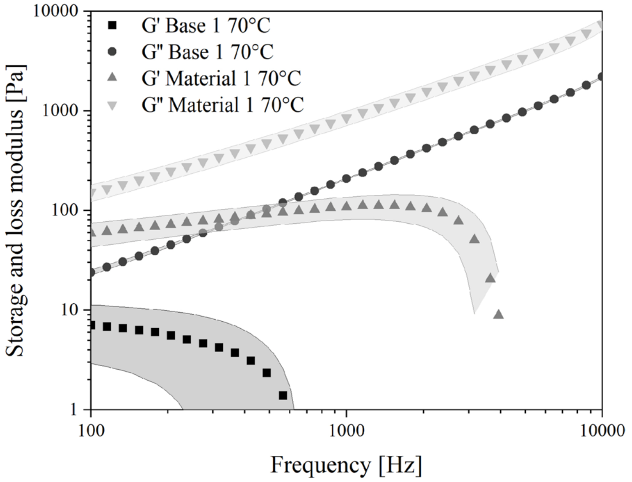

Fig. 4 Storage (G') and loss (G'') modulus of Base 1 and Material 1 at 70 °C

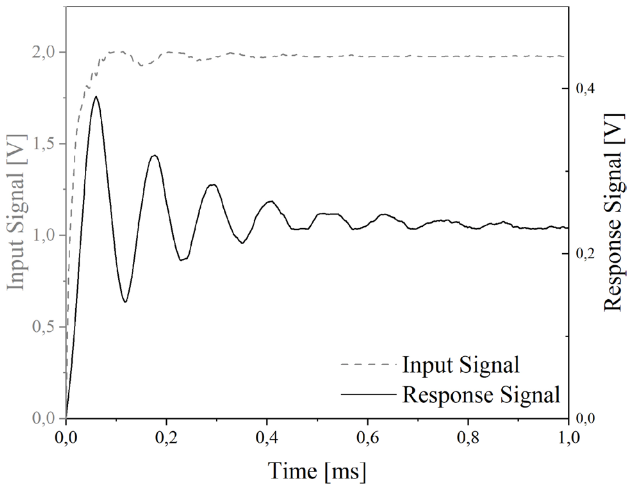

Fig. 5 Printheadmode response curve of Base 1 at 60 °C as an example

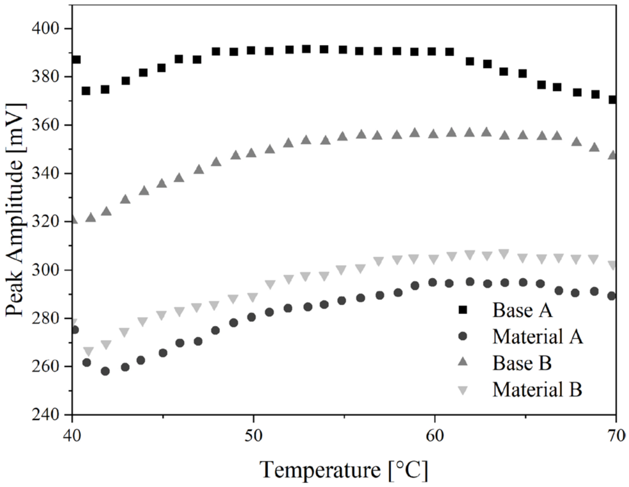

Fig. 6 Peak amplitude over temperature for Material A and B, Base A and B

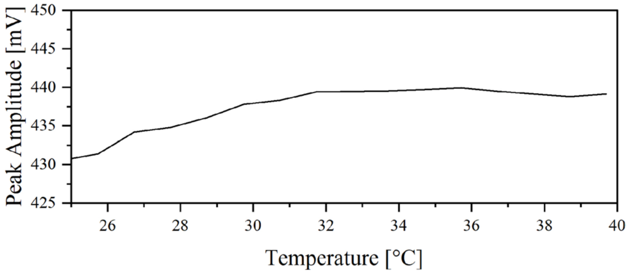

Fig. 7 Peak amplitude over temperature for PEG 400

properties relevant for jetting. The test fluids are supposed to facilitate waveform development. Heating the material for jetting is another time-consuming part, which is why a test fluid that has similar properties in a much lower temperature range is advantageous.

In this work PEG 400 was chosen as a first test fluid to represent the base A and B of the VPP resins. PEG 400 is a well-suited material for representing the Newtonian properties. While PEG 400 has a lower viscosity than the base A and B in general it has similar properties at lower temperature compared to the base at working temperature. While the base A and B have a viscosity of 60–70 mPa·s at 60 °C, PEG 400 has a similar viscosity at 30 °C. This switch in the temperature range has been made to further facilitate the measurement process. The rheological characterization of PEG 400 through the printhead mode showed that 32 °C is the temperature is most likely to be jetable which is shown in Fig. 7.

Table 1 Initial jetting parameters for PEG 400 (provided by Quantica GmbH, Berlin)

Voltage	90 V
Frequency	100 Hz
Pulse length	22 µm
Temperature	32 °C
Waveform	PND (positive–negative-drop)
Drop volume	260 pL

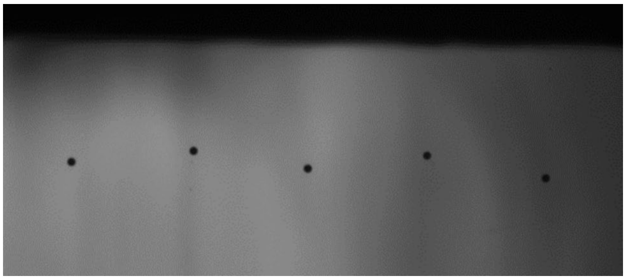

Fig. 8 Dropwatching image of PEG 400 with jetting parameters from Table 1 (captured at Quantica GmbH, Berlin)

The next step of the process in Fig. 2 is the validation. This is done through dropwatching tests of the test fluid, in this work PEG 400. Jointly with Quantica GmbH in Berlin with the NovoJet™ printhead it was shown that PEG 400 was jetable with the following first parameter set shown in Table 1. Stable droplets could be formed. This is initially in accordance with the rheological results that suggested printing at 32 °C is likely. The droplets are shown in Fig. 8 in a dropwatching image of PEG 400 with the parameters from Table 1. At the 32 °C jetting temperature PEG 400 has a viscosity of 63 mPa·s.

The last step in the workflow would be the validation of these waveform results with the base A and B of the VPP resins. Also, there is further optimization of the waveform to be done, to see whether the PEG 400 test fluid may be jetted at even lower temperatures with a higher voltage or longer pulse length.

4 Summary and outlook

Within this paper we were able to provide a systematic approach to establish requirements for test fluids from the properties of functional materials that shall be used in piezo inkjet printing. The vat photopolymerization resins and their base materials have been characterized. A test ink was chosen to represent the Newtonian base materials in first trials. A first connection between the printhead mode measurement of PEG 400 and jetting with the Quantica NovoJet™ printhead has been made by using the peak amplitude as an indicator for a suitable jetting temperature with a first waveform parameter set. Future research will show if these results can subsequently be transferred to the base materials to prove that test fluids and rheological characterization can streamline waveform development, making it more time- and cost-efficient. This principle should also be expanded to non-Newtonian materials and their corresponding test fluids. In addition, the influence of the chemical composition, in particular the molecular chain length of the monomers, on the viscoelastic behavior of the fluids on droplet formation, particle concentration, thixotropic and shear thinning properties should be investigated. These factors are likely to play a crucial role in optimizing jetting performance and ensuring reliable material deposition in more complex formulations.

Author contributions A.G. wrote the main manuscript text. J.J. prepared Fig. 1 and did the main review, as well as part of the writing. O.R provided part of the measurement data, along with A.G. All authors reviewed the manuscript.

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Data availability No datasets were generated or analyzed during the current study.

Declarations

Conflict of interest The authors declare no competing interests.

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