Data in Brief 43 (2022) 108395

Contents lists available at ScienceDirect

Data in Brief

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Data Article

Data on FTIR, photo-DSC and dynamic DSC of triethylene glycol dimethacrylate and N-vinylpyrrolidone copolymerization in the presence of ionic liquids

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ARTICLE INFO

Article history:
Received 18 May 2022
Revised 10 June 2022
Accepted 13 June 2022
Available online 25 June 2022

Dataset link: Data on FTIR, photo-DSC and dynamic DSC of triethylene glycol dimethacrylate and N-vinylpyrrolidone copolymerization in the presence of ionic liquids. (Original data)

ABSTRACT

Here we show the effect of the ionic liquid nature, its content and monomer/crosslinker ratio on the copolymerization of N-vinylpyrrolidone (NVP) with triethylene glycol dimethacrylate (TEGDMA) induced by UV or heat irradiation. For the first time, kinetics curves of photopolymerization NVP with TEGDMA in the presence of ionic liquids are obtained. The ionic liquids $EmimBF_4$, $BmimBF_4$, $OmimBF_4$ and EmimTFSI with different cation and anion structures and lengths of the alkyl radical were varied in photopolymer compositions. To understand the influence of ionic liquids on the polymerization kinetics, photo-DSC, dynamic DSC, and FTIR were performed. Parameters obtained from photo-DSC curves

DOI of original article: 10.1016/j.addma.2022.102895
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https://doi.org/10.1016/j.dib.2022.108395

Keywords:
Photopolymerization
Ionic liquid
Ionogel
FTIR
Photo-DSC
DSC

allowed to develop the methodology for the stereolithography of ionogels. The presented data constitutes the complete dataset useful for 3D-printing of ionogels with high accuracy, which is reported in the main article.

Specifications Table

Subject	Chemical Engineering
Specific subject area	photopolymerization, additive manufacturing, ionic liquids, dual curing resins.
Type of data	text file, graph, figure
How the data were acquired	Differential scanning calorimetry (DSC) was performed on Netzsch DSC214 Polyma. The FTIR spectra were acquired on the Bruker Tensor-27 spectrophotometer.
Data format	Raw Analyzed
Description of data collection	Primary data were collected via operational software with the instruments/manufacturers introductions. Data were processed using Origin 2020b 64Bit, for purposes of data analysis and diagram presentation. Dynamic DSC was performed in a nitrogen atmosphere with a heating rate of 10°C/min. Photo-DSC was performed with an irradiation intensity of 5 mW/cm^2 measured at 405 nm. The FTIR spectra were acquired in the range of 4000-400 cm^{-1} from 64 scans at a resolution of 1 cm^{-1}.
Data source location	Lomonosov Moscow State University, Moscow, Russia
Data accessibility	Repository name: Mendeley Data Data identification number: 10.17632/nd6fd96wnt.3 Direct link to the dataset: https://data.mendeley.com/datasets/nd6fd96wnt/2
Related research article	S. Nechausov, A. Ivanchenko, O. Morozov, A. Miriyev, and I. Must, "Effects of ionic liquids and dual curing on vat photopolymerization process and properties of 3d-printed ionogels," vol. 56, no. May, 2022, doi: 10.1016/j.addma.2022.102895.

Value of the Data

- These data are useful to understand the influence of ionic liquids and monomer/crosslinker ratio on photopolymerization and heat post-curing parameters of photopolymer compositions for stereolithography.
- Researchers and manufacturers can use this data in the development of new photocurable formulations for the production of ionogels used in supercapacitors, fuel cells, actuators, and batteries. Iongel DSC data is important to establish the post-curing conditions and service life of polymer materials.
- This data can be reused and combined with photopolymerization analyses of another set of formulations with ionic liquids or ionic monomers for single ionic conductivity to optimize the methods of ionogel production.

1. Data Description

The data presented in this article are related to the research article, "Effects of Ionic Liquids and Dual Curing on Vat Photopolymerization Process and Properties of 3D-printed Ionogels". The included data concerns the photopolymerization kinetics and double-bond conversion curves obtained by photo-DSC, dynamic DSC, and FTIR-spectra of a series of different compositions with

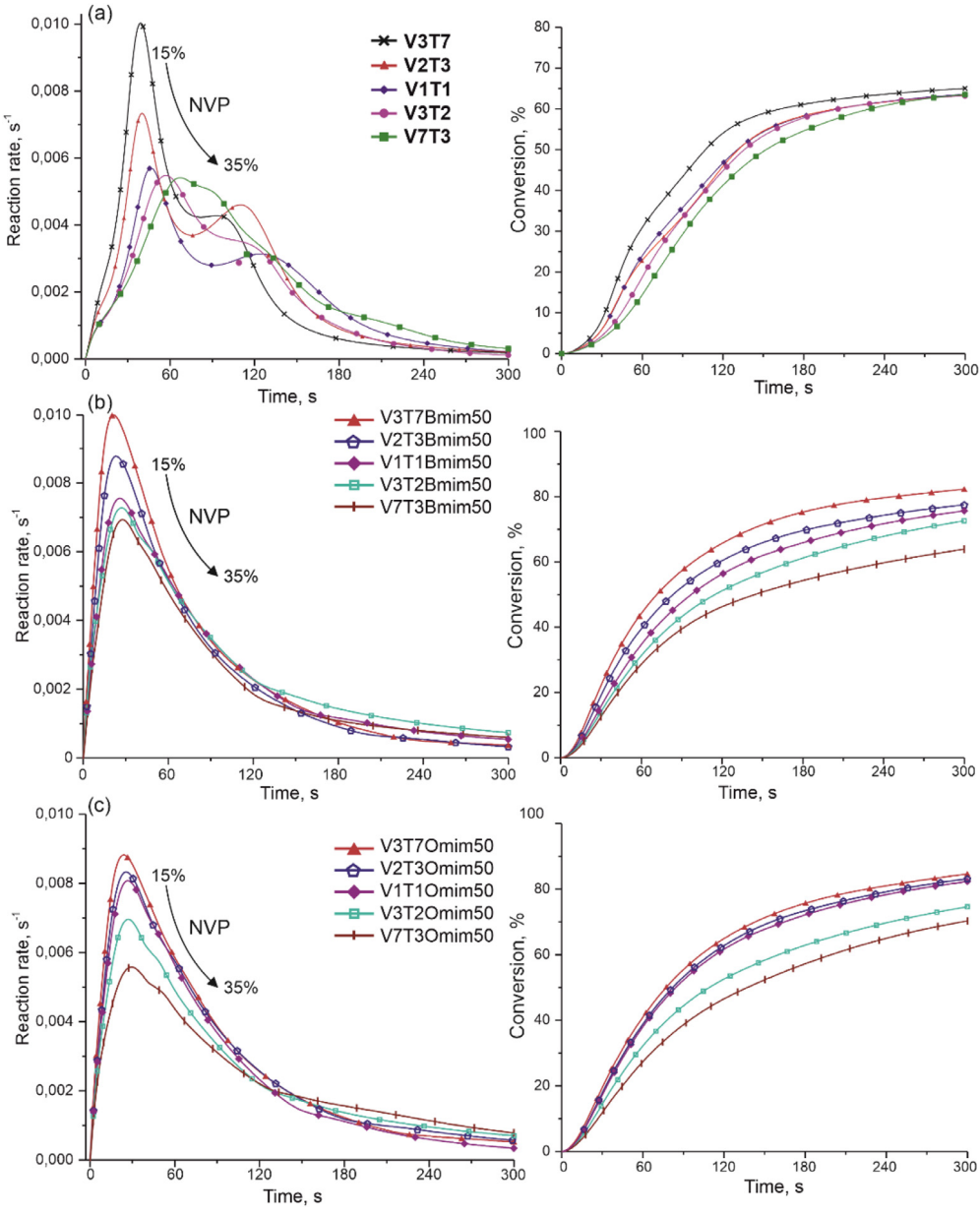

Fig. 1. Photopolymerization rate and double bond conversion in the absence (a) and the presence of 50 wt% BmimBF4 (b) and OmimBF4 (c) as a function of irradiation time for blends with different monomer/crosslinker (NVP/TEGDMA) ratios.

varied mass ratios of the crosslinker TEGDMA and the comonomer NVP as well as ionic liquid content. The mass ratios and ionic liquid content used in each composition are found in Table 2 of the companion article. In all cases, Irgacure TPO (0.5 wt% with respect to the whole composition mass) was used as a photoinitiator and AIBN (1 wt %) was used as a thermal radical iniciator. The photopolymerization rate (R_p) and double-bond conversion (p) curves are presented in Fig. 1 for variation of the monomer/crosslinker ration and in Fig. 2 for variation of the ionic

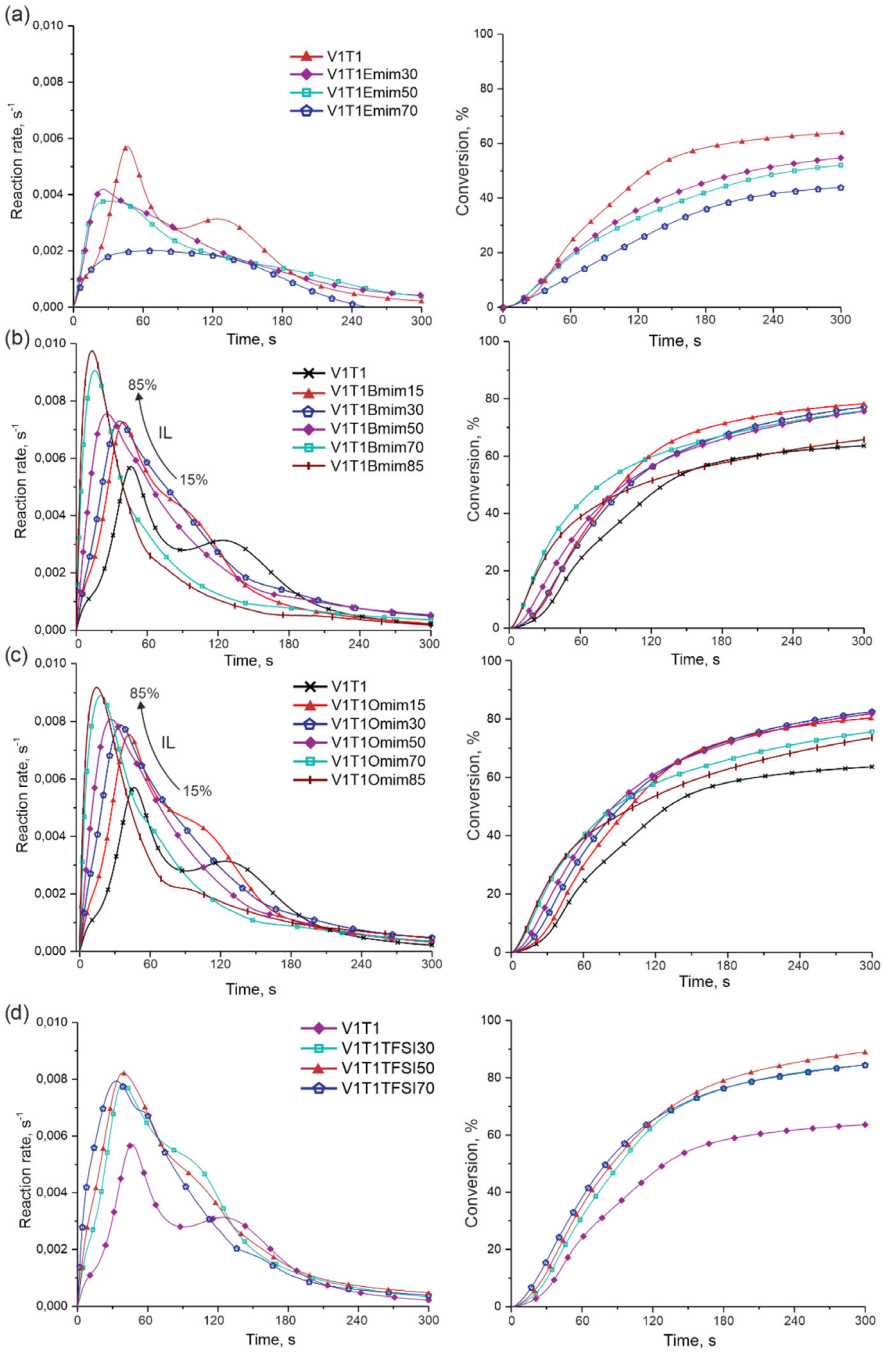

Fig. 2. Photopolymerization rate and double bond conversion as a function of irradiation time for V1T1 photocompositions with different contents of $EmimBF_4$ (a), $BmimBF_4$ (b), $OmimBF_4$ (c), or EmimTFSI (d).

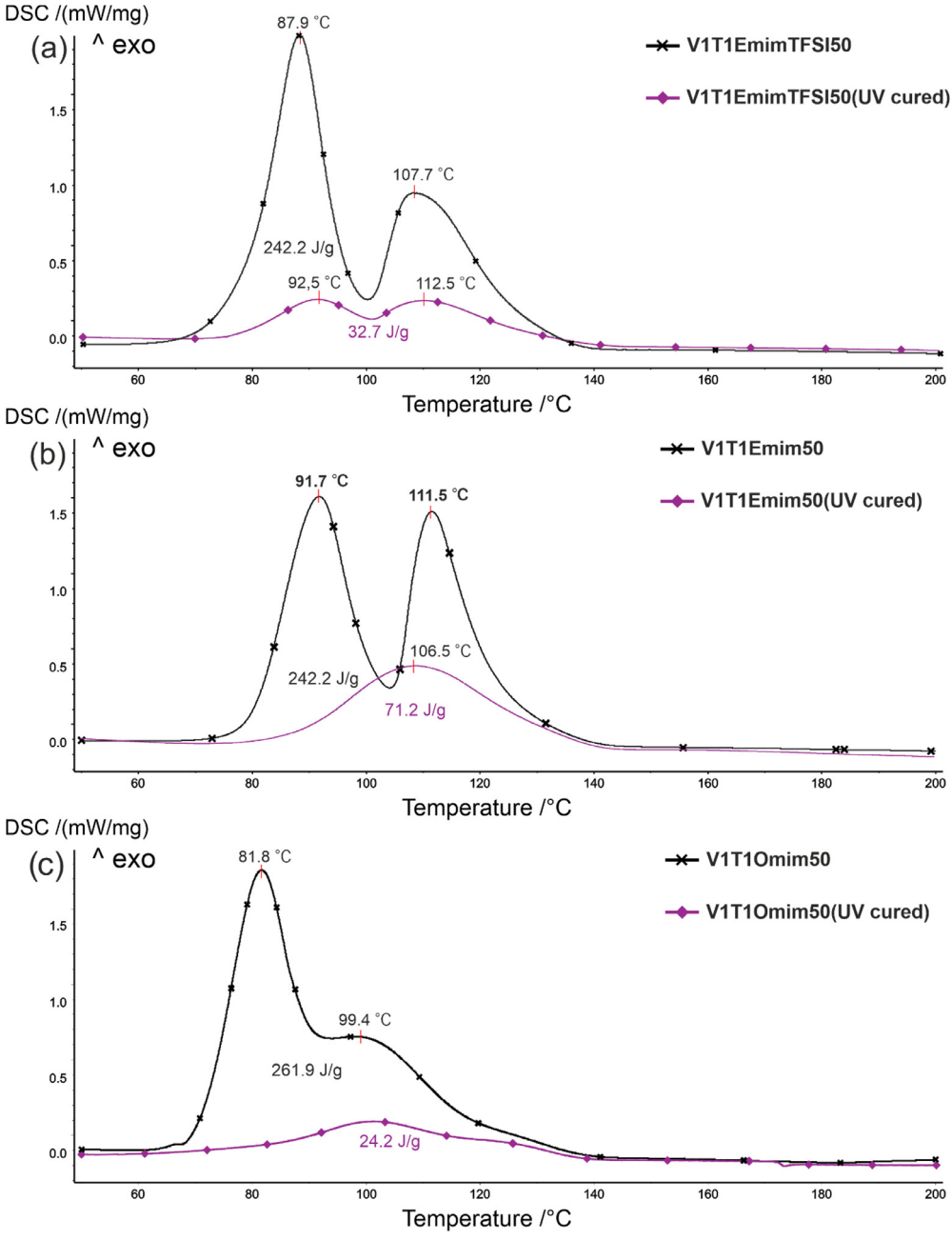

Fig. 3. DSC curves of V1T1EmimTFSI50 (a), V1T1Emim50 (b) and V1T1Omim50 (c) and their UV-cured polymers (for V1T1Bmim50 see the main article).

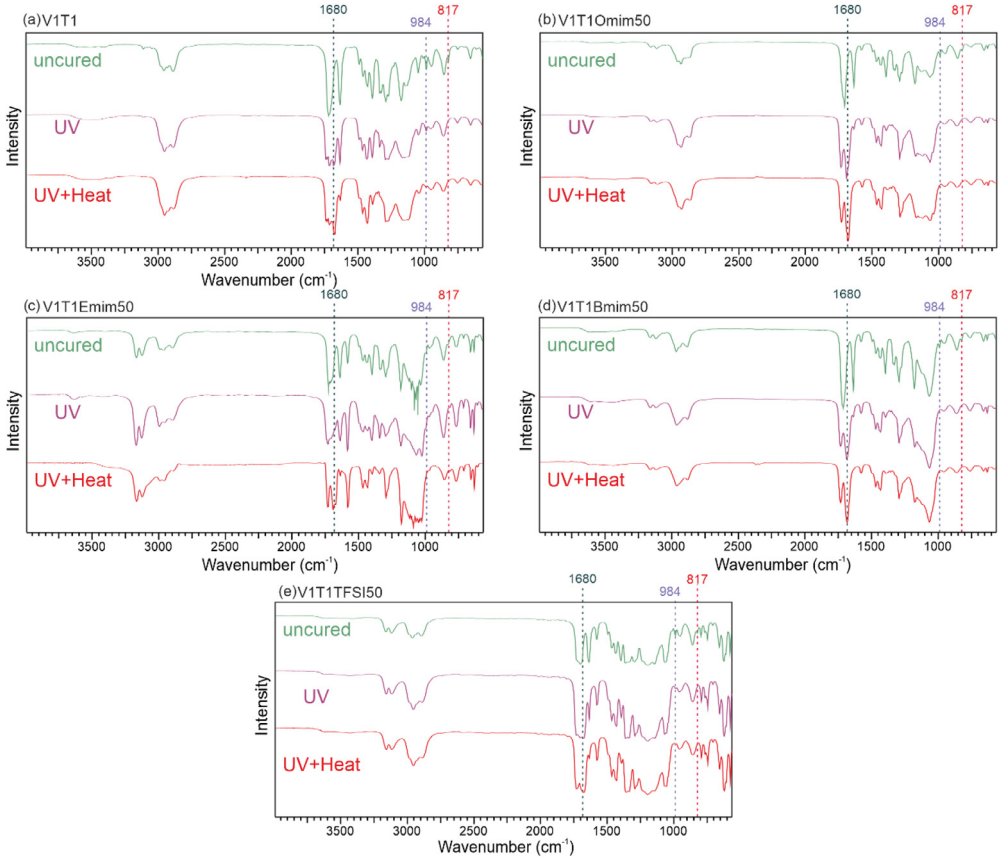

Fig. 4. FTIR-spectra of investigated photocomposition V1T1 (a), V1T1Omim50 (b), V1T1Emim50 (c), V1T1Bmim50 (d), V1T1EmimTFSI50 (e) and their 3D-printed products post-cured with UV-irradiation (purple lines) and additional heating (red lines).

liquids' content. The repository in Mendeley data contains folders with files in ASCII txt formats represented the DSC, FTIR and photo-DSC data. These txt files of the photo-DSC or DSC data contain temperature (°C) in the first column; time (s) in the second column; DSC signal (mW/mg) in the third column. The FTIR files contain wavenumber (cm^{-1}) in the first column and absorbance in the second column.

To calculate R_p and p, the standard values for the enthalpy of reaction ΔH_r for the homopolymerization of methacrylates (56 kJ mol^{-1}) [1] and NVP (53.9 kJ mol^{-1}) [2] were employed. The ΔH_r values calculated from photo-DSC were normalized based on the weight of each monomer used in the composition. Fig. 3 shows the difference in DSC curves during in heat polymerization of composition V1T1 with 50 wt% of different ionic liquids. The DSC curves of V1T1Bmim can be found in the main article.

Fig. 4 shows the evolution of FTIR-spectra in different curing steps for ionogels obtained by vat photopolymerization. For photopolymer compositions, FTIR spectrometry data is presented for both neat and post-cured with UV or heat.

2. Experimental Design, Materials and Methods

2.1. Materialsss

Triethylene glycol dimethacrylate (TEGDMA), N-vinylpyrrolidone (NVP) and Diphenyl(2,4,6-trimethylbenzoyl)phosphine oxide (TPO) were purchased from Aldrich. Azobisisobutyronitrile (AIBN) was purchased from Alfa Aesar. N-vinylpyrrolidone was distilled under vacuum to remove the NaOH inhibitor, and products of polymerization formed during storage. Ionic liquids 1-Ethyl-3-methylimidazolium bromide ([Emim]Br), 1-Butyl-3-methylimidazolium bromide ([Bmim]Br) and 1-Octyl-3-methylimidazolium bromide ([Omim]Br) were synthesized according to literature procedures [3]. Before all experiments, water content was controlled by Karl Fischer's coulometric titration (C10S, Mettler Toledo, Switzerland). Samples were dried in the presence of phosphorous pentoxide under reduced pressure (1 mm Hg) at 40 °C for 24 hours if water content was higher than 1000 ppm.

2.2. Photo-DSC data

A Photo-DSC scan was performed with a Netzsch DSC214 Polyma with a test protocol recently developed for application in vat photopolymerization analysis [4]. The DSC instrument was calibrated with a standard Indium sample. The DSC cell was equipped with a lid designed to allow the insertion of two 4.52 mm diameter fiber-optic cables (OSL2YFB, Thorlabs) connected to a UV-light source. A band pass filter centered around 405 nm was used to make the irradiation similar to that of a vat photopolymerization UV-source. A heat-absorbing filter was used to minimize the effect of the high levels of thermal radiation generated by the photocuring source (OSL2, Thorlabs, 150 W, 3200 K). The intensity of irradiation in photo-DSC experiments was measured using the UV-radiometer "TKA-PKM" equipped with a UV-A or 405 nm sensor ("TKA Scientific Instruments", Russia). A standard 40 μl aluminum crucible (Netzsch) with an inner bottom diameter of 5.07 mm was used without the cover lid. 2.8 mg of the sample was spread over the inner surface of the crucible and placed in the DSC-cell. The DSC chamber was purged with argon for 30 min at a rate of 40 mL/min prior to irradiation in order to remove oxygen from the sample before each experiment. The final conversion was measured after 600 seconds of irradiation. The experiment for each sample was performed 3 times that were differ in maximum polymerization rate and in maximum conversion not more than in 3%. Each photopolymerization curve was built as average result of 3 runs.

2.3. Dynamic-DSC data

Differential scanning calorimetry (DSC) was performed on Netzsch DSC214 Polyma at a heating rate of 10°C/min in the nitrogen atmosphere.

2.4. FTIR data

FTIR study was performed between two KBr windows. The uncured photopolymer composition was applied to the KBr window, then pressed together with the 100 micrometer PP film by another window. After obtaining the FTIR-spectrum of the uncured composition, it was photopolymerized with a UV-lamp for 2 min with a light intensity of 25 mW/s (UV). Then it was additionally thermally polymerized in an oven using a heating rate of 10°C/h up to 80°C during 6 h (UV+Heat). It is worth noting that it was not possible to photopolymerize samples between ZnSe windows due to their opacity in the UV-region.

Ethics Statements

This work did not involve human subjects, animal experiments, or data collected from social media platforms.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

Data Availability

Data on FTIR, photo-DSC and dynamic DSC of triethylene glycol dimethacrylate and N-vinylpyrrolidone copolymerization in the presence of ionic liquids. (Original data) (Mendeley Data).

Acknowledgments

The reported study was performed under the «ERA.Net RUS plus» program and funded by RFBR, project number 20-53-76021. Research at Empa was performed under the «ERA.Net RUS plus» program, funded in Switzerland by the Swiss National Science Foundation, grant number IZRPZ0_194986. Research at RTU was performed under the ERA-NET program funded by VIAA, project UPRINTAROBOT Nr ES RTD/2021/8. Research at TU was supported by the «ERA.Net RUS plus» program MOBERA26 and Estonian Research Council grants PRG1498 and PRG1084.

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