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Data in Brief

Data Article

Critical overlap concentration and intrinsic viscosity data of xanthan gum aqueous solutions in dimethyl sulfoxide

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A R T I C L E   I N F O

A B S T R A C T

This is the supplementary information of the research paper "Haemodynamics around confined microscopic cylinders" by Rodrigues et al. [1]. The critical overlap concentration of entanglement of polymer coils indicates whether a polymer solution is dilute or semidilute. Here, the reader will find the determination of $c*$ for xanthan gum aqueous solutions in 52 wt.% of dimethyl sulfoxide, often used as non-particulate blood analogues. From the shear flow curves of a dilution series of the polymer the zero-shear viscosities η_0 were obtained, allowing us to estimate the intrinsic viscosity $[\eta]$ based on the xanthan gum concentration of the fluids. Two methodologies for doing so are described: using information from multi-concentration measurements and from a single polymer solution (rough estimate). With the intrinsic viscosity the determination of $c*$ is straightforward.

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Specifications Table

Subject	Chemical Engineering (General)
Specific subject area	Polymer Rheology
Type of data	Table
	Figure
How data were acquired	Instruments: stress-controlled shear rheometer
	Make and model and of the instruments used: Anton Paar Physica MCR 301
Data format	Raw
	Analyzed
Parameters for data collection	Xanthan gum aqueous solutions in 52 wt.% of dimethyl sulfoxide were prepared with different polymer concentrations and characterised over a shear rate range of about 0.0025–25100 s^{-1} using a stress-controlled rotational rheometer, as explained in Rodrigues et al. [1]. With the information gathered from the rheological characterisation a series of calculations were performed in order to estimate the intrinsic viscosity and critical overlap concentration.
Description of data collection	With the composition and flow curves (obtained by shear rheometry) of two polymer solutions from a dilution series of xanthan gum in an aqueous solution of dimethyl sulfoxide, the intrinsic viscosity was determined using information (1) from both and (2) from a single fluid (distinct methodologies). The critical overlap concentration of entanglement was then estimated. Additional insight into the mathematical formulae used can be attained via the literature cited throughout the text.
Data source location	Institution: Centro de Estudos de Fenómenos de Transporte (CEFT), Departamento de Engenharia Química, Faculdade de Engenharia da Universidade do Porto; City/Town/Region: Porto; Country: Portugal
Data accessibility	With the article
Related research article	T. Rodrigues, F.J. Galindo-Rosales, L. Campo-Deaño, Haemodynamics around confined microscopic cylinders, J. Non-Newton. Fluid Mech. 286 (2020) 104406. https://doi.org/10.1016/j.jnnfm.2020.104406

Value of the Data

- Xanthan gum-based aqueous solutions are often used as a non-particulate whole blood replacement for *in vitro* experiments. Gathering data of their rheological properties is important for a complete characterisation of the blood analogues. The critical overlap concentration c^* is what sets apart dilute from semidilute polymer solutions, hence learning the alternatives for estimating the intrinsic viscosity $[\eta]$ and subsequently c^* is useful and the methods are widely applicable.
- Polymer rheologists can benefit from these data, as they typically seek to characterise a variety of polymer solutions for numerous applications.
- The methodologies described instruct the reader on how to determine the intrinsic viscosity and estimate the critical overlap concentration for any polymer solution (polymer dissolved in solvent). Being a characteristic value of xanthan gum aqueous solutions in 52 wt.% of dimethyl sulfoxide, the critical overlap concentration data can be used as is to characterise the same mixture regardless of the polymer concentration (slightly different concentrations of dimethyl sulfoxide in water are not expected to affect c^* and $[\eta]$ substantially).
- Knowing if a polymer solution is dilute or semidilute is important for analysing its uniaxial extensional flow dynamics. Generically, the more dilute a solution is, the harder it will be to perform such analysis [2].

1. Data Description

Determination of the critical overlap concentration of entanglement c^* and intrinsic viscosity $[\eta]$ for xanthan gum aqueous solutions in 52 wt.% of dimethyl sulfoxide. Using information from the flow curves of what is quintessentially a dilution series of xanthan gum (see Rodrigues et al.
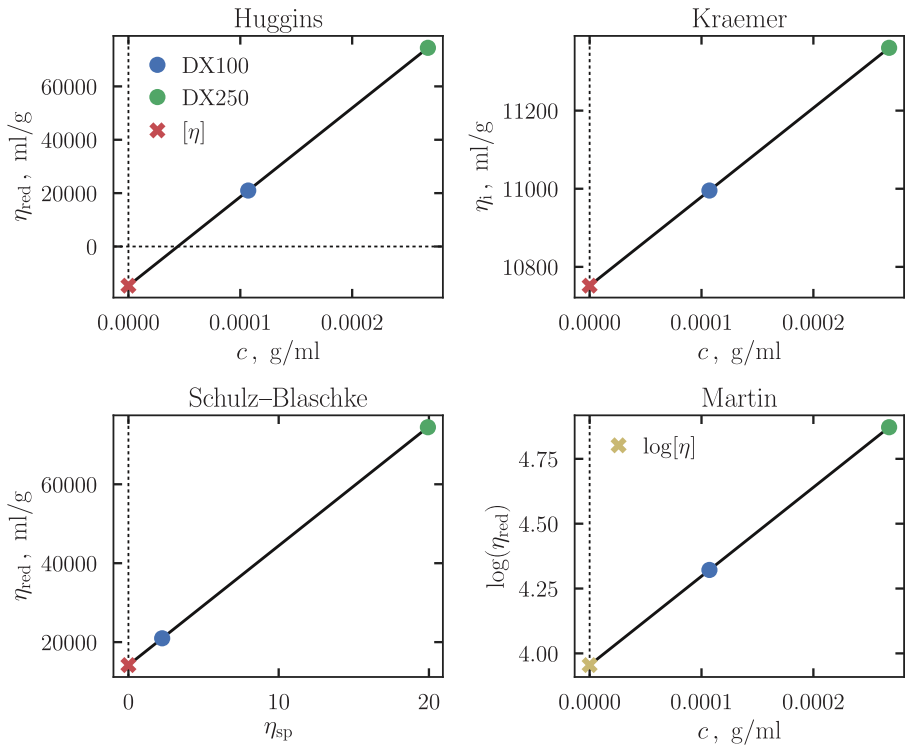
Fig. 1. Determination of the intrinsic viscosity from multi-concentration measurements.

Table 1
Properties of the two solutions considered from the xanthan gum dilution series.

	c [g/ml]	η_0 [mPa·s]	η_r	η_{red} [ml/g]	η_i [ml/g]	η_{sp}
DX100	1.1×10^{-4}	9.87	3.25	20973.9	10995.4	2.25
DX250	2.7×10^{-4}	63.6	20.9	74456.5	11360.8	19.9

Table 2
Intrinsic viscosity and critical overlap concentration data.

	Kraemer	Schulz–Blaschke	Martin	Solomon–Ciuta
$[\eta]$ [ml/g]	10752	14182	9014	13652
c^* [g/ml]	1.3×10^{-4}	1.0×10^{-4}	1.6×10^{-4}	1.1×10^{-4}

[1]), two methodologies based on multi- and single-concentration measurements are described to estimate the just-mentioned critical concentration. Fig. 1 depicts how to perform the determination of $[\eta]$ from multi-concentration measurements. Table 1 gathers the rheological properties of the polymer solutions required to estimate, $[\eta]$ whose values are presented in Table 2 alongside the critical overlap concentration calculated according to each method. The supplementary spreadsheet lists the results presented throughout the text and explains how these were obtained. There, the reader will find the properties of the polymer solutions, the slope and *y*-intercept that yield from the linear fits of the data and the critical overlap concentration calculated from these values.

2. Experimental Design, Materials and Methods

The critical overlap concentration of entanglement c^* of polymer coils is one of the most important characteristic values of a polymer solution [2]. As the concentration c of polymer coils in a solution increases, these begin to interact and entangle. c^* sets the upper limit that defines a dilute solution ($c/c^* \leq 1$) *i.e.* a solution in which the polymer coils still have sufficient space to avoid any interactions. For $c > c^*$ (or $c/c^* > 1$) the polymer solution becomes semidilute and the coils begin to entangle, interact and contract with increasing concentration.

c^* can be estimated directly from the intrinsic viscosity $[\eta]$ using the expression [3]:

$$c^* \sim \frac{1.45}{[\eta]}$$

which considers the ideal volume requirement $4\pi R_g^3/3$ of a polymer coil, where the radius of gyration R_g is obtained from the Flory–Fox equation (xanthan gum's molecular weight is $M_w^{XG} \sim 10^6$ g/mol) [4,5]:

$$R_g = \left(\frac{[\eta]M_w^{XG}}{\Phi_0} \right)^{1/3}$$

where $\Phi_0 = 3.67\times10^{24}$ mol^{-1} is the Flory constant.

Calculating, $[\eta]$ however, is more complex. First it was determined experimentally from multi-concentration measurements with the first two terms of the Taylor-series:

$$\eta_{\text{red}} = \frac{\eta_{\text{sp}}}{c} = [\eta] + k[\eta]^2 c + \dots$$

where η_{red} is the reduced viscosity, η_{sp} is the specific viscosity and k is a coefficient. A linear dependency of the reduced viscosity η_{red} from the concentration c is evident, with $a = k[\eta]^2$ being the slope and $b = [\eta]$ the y-intercept ($\eta_{\text{red}} \sim b + ac$). The intrinsic viscosity is then determined from the y-intercept of a plot of η_{red} *versus* c by extrapolating the linear fit of the data to $c \to 0$ *i.e.* to infinite dilution. The previous equation can be further developed taking into consideration the following definitions:

$$\eta_{\text{sp}} = \eta_{\text{r}} - 1 \sim \frac{\eta_0}{\eta_s} - 1$$

η_{r} being the relative viscosity, η_0 the zero-shear viscosity and η_s the viscosity of the solvent ($\eta_s^{D52} \sim 3.04$ mPa·s).

The multi-concentration measurements correspond to a dilution series of the polymer, which consists of the xanthan gum aqueous solutions in dimethyl sulfoxide prepared and characterised by Rodrigues et al. [1]. For an "exact" analysis the relative viscosities η_{r} of the polymer solutions should lie between 1.2 and 2.5 [6]. For DX100 and DX250, the two least concentrated solutions—hence those with the lowest η_0 and subsequently η_{r}—these are 3.2 and 20.9, respectively (Table 1). This means that, ideally, solutions of even lower polymer concentration c (and more than two, if possible) should be used in order to increase the precision of the c^* estimate. Nevertheless, as far as an estimate goes, using DX100 and DX250 works just fine.

Various regression models based on the Taylor-series introduced above can be used to perform the linear extrapolation, depending on the type of polymer [6,7]:

- Huggins:

$$\eta_{\text{red}} \sim [\eta] + k_{\text{H}}[\eta]^2 c$$

- Kraemer:

$$\eta_{\text{i}} \sim [\eta] + k_{\text{K}}[\eta]^2 c$$

  where $\eta_{\text{i}} = \ln(\eta_{\text{r}})/c$ is the inherent viscosity;
- Schulz–Blaschke:

$$\eta_{\text{red}} \sim [\eta] + k_{\text{SB}}[\eta]\eta_{\text{sp}}$$

- Martin:

$$\log(\eta_{\mathrm{red}}) \sim \log[\eta] + k_{\mathrm{M}}[\eta]c$$

The Kraemer, Schulz–Blaschke and Martin equations all led to similar c^* values. With the Huggins equation, however, the extrapolation of the reduced viscosity to zero concentration (see Fig. 1) resulted in an unrealistic $[\eta]$ value (negative). Note, though, that the condition $1.2 < \eta_{\mathrm{r}} < 2.5$ is not respected and the Huggins formulation might be more sensitive to this, being best suited for less concentrated dilution series. Besides reducing the polymer concentration c of the solutions, using one more term in the Taylor-series—which becomes a second-degree Taylor polynomial—is suggested in order to obtain a sound result with the Huggins equation as well.

It is also possible to calculate the intrinsic viscosity $[\eta]$ using information from only one polymer solution. Although not as accurate as the previous method—that uses multi-concentration measurements—it serves the purpose of a rough estimate. Several equations do the trick but here the Solomon–Ciuta equation was used because of its simplicity [8]:

$$[\eta] \sim \frac{\sqrt{2\left[\eta_{\mathrm{sp}} - \ln(\eta_{\mathrm{r}})\right]}}{c}$$

For DX100 (the solution with lowest c) a critical overlap concentration of entanglement c^* similar to those determined via the multi-concentration measurements approach was obtained.

Ultimately, the averaged critical overlap concentration—determined via the Kraemer, Schulz–Blaschke and Martin extrapolations, and the Solomon–Ciuta equation—was approximately 0.012 wt.% $\sim$ 0.021 g of XG $\sim$ 1.3×10^{-4} g/ml (Table 2).

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships which have, or could be perceived to have, influenced the work reported in this article.

Acknowledgments

Appendix A. Supplementary Material

Supplementary material associated with this article can be found in the online version at doi:10.1016/j.dib.2020.106431

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