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Investigation on the Flow Behaviour of Agar Solution and Rheological Modelling

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ABSTRACT

In this study, the effect of shear rate, agar concentration (15-35 kg/m^3) and temperature (30-60°C) on the apparent viscosity of the agar solution was investigated. Apparent viscosities at different shear rates were determined using a rotational viscometer. The findings show that the apparent viscosity decreasing with a rise of shear rate increased with increasing concentration. An increase in the tempearture, on the other hand, led to a decrease in the apparent viscosity. To illustrate both temperature and concentration changes together, the Bingham, power law, and Casson models were utilized in choosing the most suitable model. When the models were compared using statistical tests, the most compatible model was found to be the power-law model. The consistency coefficient and flow behaviour index calculated using the power law model showed that agar solutions exhibited shear-thinning flow behavior (pseudoplastic). This study suggests that power law, within the ranges of the temperature and concentration studied could be used to estimate the viscosity of the agar solutions in the applications requiring the knowledge of flow behavior.

Keywords: Flow behaviour, Rheological models, Agar-agar, Rotary viscometer.

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Introduction

Agar is linear polysaccharide consisting of β-D-galactopyranose and 3,6-anhydro-α-L-galactopyranose repeating units. Used in food, pharmaceutical and biotechnological applications due to its unique physico-chemical properties and gelling properties, agar is a valuable hydrocolloid found in red seaweed species. It is found as intercellular matrix material in many red seeweed species. Agar is an extract from some red algae (Gelidiacae, Sphaerococcaceae and Rhodophyceae) and contains agarose and agaropectin polysaccharides. Gelling of the agar is due to the agarose polysaccharide contained in it, however, it is the agaropectin fraction providing the viscosity [1-5].

Food, pharmaceuticals, cosmetics, microbiology and biotechnology, textiles and dentistry are some of the industries where agar-agar finds widespread use due to its unique properties and versatility. It is widely used as a vegetarian alternative to gelatin, especially in the food industry due to its gelling properties. It also acts as a thickening agent in soups and sauces, helping to give food a smooth and consistent texture without altering its flavor. Other common uses of agar-agar in food are as a stabilizer in food products to prevent component separation or precipitation, and as a nutrient culture medium for growing microorganisms for scientific research in laboratories or for the production of probiotics and other microbial products [2, 6, 7]. Agar is advantageous over other gelling means as it can form a stable gel at low concentrations and over a wide pH range [6-9]. In studies related to the gelation of agar, the transition temperature from the solution to gelation was reported to be 45 ^{o}C [10]. Also, the agar solution gelation is reversible as it is a physical gelation caused only by hydrogen bridges [11].

Determining the rheological properties of agar solutions is crucial for quality control, process optimization, formulation development, product stability, scaling production processes, and advancing scientific research in various industries. By characterizing the rheology, information is gained about the consistency, texture and flow behavior of agar solutions and manufacturers can ensure the quality and consistency of their agar products. Also, changes in viscosity, gel strength, or flow behavior over time may indicate degradation or aging of agar solutions, affecting their functionality and performance. Rheological data help establish storage conditions and determine appropriate expiration dates for agar-based products. Aqueous agar solution that do not comply rheologically with Newton's viscosity law is typical a Non-Newtonian fluid [9]. Many investigations have been done on the effects of shear rate, temperature, concentration, and molecular weight parameters on the apparent viscosity of agar aqueous solutions in the literature. [8, 12-15].

The apparent viscosity of agar-agar varies depending on factors such as its specific source and grade, the extraction method, and the experimental conditions used in the studies. This study aimed to provide critical information for product development, process optimization, quality control, stability, scale-up and basic research evaluation by investigating the effects of temperature, concentration and shear rate on the rheological behavior of agar solutions as well as the combined effects of temperature and concentration on apparent viscosity.

Materials and Methods

Material

Agar was purchased from Smart Chemistry (Izmir, Turkey), a local producer. De-ionized water was used to prepare all solutions.

Preparation of Agar Solution at Different Concentrations

Firstly, 3, 4, 5, 6 and 7 g of agar were weighed and dissolved in 200 ml of distilled water to prepare agar solutions of different concentrations. In order for the agar to dissolve easily and the prepared solution to be homogeneous, the agar was slowly added to the distilled water heated to 90 °C. In order to remove air bubbles in the solution, which was stirred for 24 hours on a magnetic stirrer, the mixture was left at room temperature for 6 hours. The mass concentrations of the agar solutions prepared are 15, 20, 25, 30 and 35 kg / m^3, respectively.

Measuring the Apparent Viscosity of Agar Solution

Apparent viscosities were determined using a rotational viscometer. A Brookfield viscometer (model LVDV-E; Brookfield Engineering Laboratories. Inc., Stoughton, MA, USA) with spindle speeds of 10, 12, 20, 30, 50, 60 and 100 rpm was used.The water-jacketed stainless steel cylindrical vessel was utillized for all measurements. Approximately 150 ml of sample was filled into the cylindrical vessel with a volume of 200 ml for each measurement. The water-jacketed cylindrical vessel was connected to a temperature bath to maintain the temperature constant at the desired value. The shear rate was gradually increased from 1.32 to 22 s^{-1}. The torque measurements were performed and each measurement was repeated twice for reliability of the results.

Shear rates for agar solutions were calculated by utilizing the cylindrical spindle factors given by Brookfield (Brookfield Engineering Labs. Inc., Stoughton, MA, USA) for spindle LV-1.

$$-dV_{\varphi}/dr=0.22N \quad (1)$$

where $-dV_{\varphi}/dr$ (s^{-1}) is the shear rate, N is the spindle speed (rpm).

The viscometer was calibrated using the Brookfield silicone viscosity standard (nominal viscosity at 25 ° C: 1000 mPa s). The following equation was used to calculate spindle calibration factor for any spindle speed.

$$F=R/100 \quad (2)$$

where f is the spindle calibration factor, R is the full scale viscosity range.

The following equation was used to calibrate the apparent viscosity values (η_a).

$$\eta_a=f\ (\%\text{Tork}) \quad (3)$$

Equation 4 was used to calculate the shear stress.

$$\tau_{r\phi}= \eta_a\,(-dV_{\phi}/dr) \quad (4)$$

where $\tau_{r\phi}$ is the shear stress (mPa),η_a is the apparent viscosity (mPas), dV_{ϕ}/dr is the shear rate (s^{-1}), V_{ϕ} is the velocity in direction along the axis of rotation, r is the radial distance.

Determination of the Rheological Model

Flow curves for agar solutions were created by applying experimental data to power-law, bingham and casson models, which are the most commonly used models of Non-Newtonian models [12, 16-18]. The mathematical expression of these models is as follows:

$$\tau_{r\phi}= m\,(-dV_{\phi}/dr)^n \quad \text{(Power Law Model)} \quad (5)$$

$$\tau_{r\phi}=\tau_B + \eta_B\,(-dV_{\phi}/dr) \quad \text{(Bingham Model)} \quad (6)$$

$$\tau_{r\phi}^{\ 0.5}=\tau_C + \eta_C\,(-dV_{\varphi}/dr)^{0.5} \quad \text{(Casson Model)} \quad (7)$$

where m is the consistency index (mPas) and n is the flow behaviour index (dimensionless), τ_B and η_B are the Bingham model parameters, τ_C and η_C are the Casson model parameters.

The model parameters were found by applying non-linear regression to these model equations at different temperatures and concentrations, and the highest correlation coefficient was used to determine the model that best fits the experimental data.

Statistical Analysis

The statistical software package (Statistica for Windows 5.0, 1995) was used to perform nonlinear regression analysis of experimental viscosity data of agar solutions. Variance analysis (ANOVA) was used to evaluate the effect of concentration on flow activation energy and consistency index statistically.

Results and Discussion

Determination of the Rheological Model

Model parameters of Power-law (Ostwald-de Waele), Bingham and Casson models, which are frequently used in engineering applications [18-20], were determined by nonlinear regression analysis for different temperatures and concentrations, and these values are given in Table 1.

Table 1. Model parameters and correlation coefficients of rheological models for agar solution at different temperatures and concentrations

T (°C)	C (kg/m³)	Power law model			Bingham model			Casson model		
		n	m (Pas^n)	r^2	η_B (Pas)	τ_B (Pa)	r^2	η_C (Pas)$^{0.5}$	τ_C (Pa)$^{0.5}$	r^2
30	15	0,4262	0,2491	0,9799	0,028	0,3275	0,8732	0,1166	0,4286	0,9344
	20	0,5199	0,4173	0,9882	0,0727	0,5402	0,9371	0,2049	0,5024	0,9655
	25	0,5401	0,6386	0,9992	0,1294	0,7623	0,9827	0,278	0,5833	0,995
	30	0,5201	0,8847	0,9906	0,1682	1,044	0,9797	0,3098	0,703	0,9857
	35	0,6468	1,1911	0,9979	0,028	0,3275	0,8732	0,498	0,6844	0,9927
40	15	0,2216	0,2277	0,9948	0,0101	0,258	0,9447	0,0509	0,4471	0,9845
	20	0,5046	0,3656	0,9921	0,064	0,4431	0,9704	0,1892	0,4633	0,9838
	25	0,5318	0,5883	0,9966	0,1123	0,7264	0,9676	0,2572	0,5748	0,9857
	30	0,5109	0,8101	0,9812	0,1479	0,9648	0,9642	0,288	0,6825	0,9718
	35	0,6863	0,9692	0,9974	0,3282	1,1135	0,9837	0,492	0,5773	0,9912
50	15	0,2908	0,1742	0,9962	0,0117	0,2021	0,9618	0,0626	0,3769	0,9923
	20	0,4086	0,3227	0,9779	0,0395	0,3771	0,9737	0,1356	0,4674	0,9868
	25	0,5547	0,4559	0,9939	0,0992	0,5329	0,9786	0,2474	0,4767	0,993
	30	0,5353	0,6579	0,9923	0,1299	0,7935	0,9745	0,2768	0,6001	0,9851
	35	0,6143	0,8465	0,994	0,2121	1,0863	0,9544	0,3782	0,6321	0,9775
60	15	0,1606	0,1597	0,9824	0,0044	0,1771	0,8868	0,0286	0,3854	0,9455
	20	0,4291	0,2433	0,9617	0,032	0,2895	0,9608	0,1252	0,4005	0,9761
	25	0,5265	0,3998	0,9867	0,0763	0,4855	0,9566	0,2131	0,4668	0,9842
	30	0,534	0,5772	0,9913	0,117	0,6727	0,98	0,2641	0,5487	0,9934
	35	0,575	0,7667	0,999	0,1734	0,9338	0,9767	0,3313	0,6188	0,9911

According to the data in Table 1, since the correlation coefficient (r^2) is higher than the other models, the power-law model is the model that best expresses the experimental data. The power law model estimating the apparent viscosities of polymer solutions is frequently encountered. Rahimi and Dehaghani (2021) developed a viscosity model based on the power law rheological model to estimate the viscosity of polymer solutions in their experimental study on a novel correlation predicting the apparent viscosity of sulfonated polyacrylamides. In another study, power law was used to generate flow curves from the rheological data of the water-carboxymethyl cellulose-salt system [22].

Flow behavior index (n) and consistency index (m) are important parameters that determine non-newtonian flow properties. In this model, it is seen that the flow behavior index is less than 1, so the flow behavior of agar solutions is pseudoplastic (shear thinning behavior). That is, the apparent viscosity decreases sharply at low shear rates, while this change is less at high shear rates. This indicates less resistance to flow due to the disorder of the long chain molecules [16].

The apparent viscosity of agar solutions in the concentration range of 15-35 kg/m^3 as a function of shear rate at constant temperature (40 °C) is shown graphically in Figure 1. An increase in the shear rate of the agar solutions resulted in a reduction in apparent viscosity.

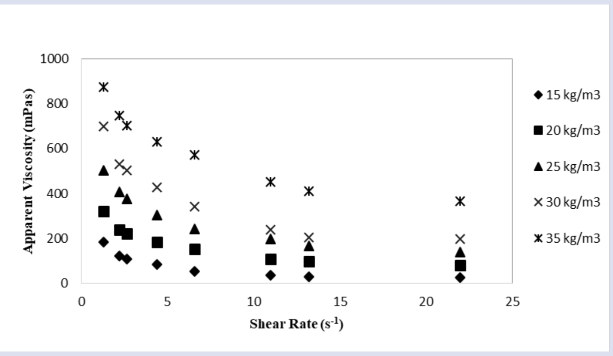

Figure 1. Typical flow curves for different concentrations of agar solutions at 40 ^{0}C.

Figure 1. shows that the apparent viscosity increases with increasing concentration. As the interaction between agar particles decreases at lower concentrations, apparent viscosity decreases compared to higher concentrations [12]. The non-Newtonian behavior of agar-agar solutions is attributed to the entanglement of the agarose chains and the formation of a three-dimensional network structure. Shear thickening behavior at low shear rates was associated with increased resistance to flow due to the entangled mesh. As the shear rate increased, the network began to deteriorate, causing shear thinning and a decrease in viscosity [23].

Effect of Temperature on the Rheological Behavior of Agar Solution

Since the agar solution is exposed to different shear rates in industrial applications, the equipment used can be better designed depending on the variation of viscosity with temperature at determined shear rates. It was determined that the apparent viscosity of the agar solutions decreased with increasing temperature. At lower temperatures, agar agar molecules are in a helical structure, which allows them to form a three-dimensional network, trapping water and creating a gel-like consistency. This gel network gives agar agar its high apparent viscosity. As the temperature increases, the heat energy disrupts the helical structure, causing the agar agar molecules to unwind and the gel network to weaken. At higher temperatures, the helical structure is completely disrupted, and the agar agar molecules exist in a random coil conformation. In this state, the agar agar molecules can move more freely, and the viscosity decreases. The breakdown of the gel structure leads to a decrease in the apparent viscosity of the agar agar solution [24]. The effect of temperature on the viscosity of polymer solutions is expressed by the Arrhenius equation and the equation is as follows:

$$\eta_a = Ae^{Ea/RT} \tag{8}$$

where η_a is the apparent viscosity (mPas), A is a constant (mPas), E_a is the activation energy of flow (kJ/mol), R is the universal gas constant (8.314×10^{-3} kJ/mol.K), T is the absolute temperature (K).

Flow activation energy is a measure of temperature sensitivity that is directly related to the size of the macromolecule and determines the degree of difficulty of the flow. The Arrhenius plot drawn for a given shear rate (11 s^{-1}) is shown in Figure 2.

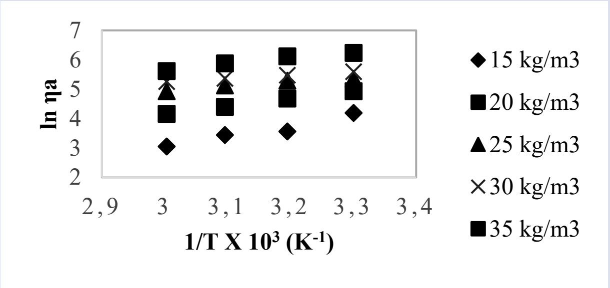

Figure 2. Arrhenius plot for different concentrations at shear rate 11 s^{-1}.

The maximum and minimum values for the different shear rates of the flow activation energies calculated from the slopes of the lines obtained by plotting ln η_a versus 1/T for different concentrations are shown in Table 1.

Considering the maximum values of flow activation energy, it has been found that the viscosity of agar solutions is relatively more sensitive to temperature change at high shear rates.

Table 2. The change of flow activation energy of agar solutions at different shear rates.

Shear rate (s^{-1})	**Ea (kJ/mol)**	**r^2**
1.32	9.906-14.36	0.9583-0.9862
2.20	12.44-19.25	0.9686-0.9933
2.64	12.06-20.68	0.9610-0.9989
4.40	13.49-23.56	0.9441-0.9960
6.60	14.70-27.14	0.9183-0.9707
11.0	9.509-29.95	0.9405-0.9995
13.2	7.558-28.76	0.9268-0.9973
22.0	12.08-30.12	0.9226-0.9891

Combined Effect of Temperature and Concentration on the Apparent Viscosity

The ability to derive a single equation that describes the combined effects of temperature and concentration on the apparent viscosity of agar solutions would be extremely useful in a variety of applications. The change of apparent viscosity with temperature can be written in two ways:

$$A = \delta(C)^{\varepsilon} \tag{9}$$

$$A = \delta_1 \exp(\varepsilon_1 C) \tag{10}$$

By substituting these equations in the Arrhenius equation, the following equations are obtained, which give the effect of temperature and concentration on the apparent viscosity of agar solutions.

$$\eta_a = \delta(C)^{\varepsilon} \exp(E_a/RT) \tag{11}$$

$$\eta_a = \delta_1 \exp(\varepsilon_1 C)\exp(E_a/RT) \tag{12}$$

Table 3. The change of agar solution to apparent viscosity with temperature and concentration (Eq. 11)

Shear rate (s^{-1})	**$\eta_a = \delta(C)^{\varepsilon} \exp(E_a/RT)$**			
	δ mPas(kg/m³)$^{-\varepsilon}$	**ε [–]**	**E_a (kJ/mol)**	**Statistical tests**
1.32	0.0129	1.854	11.84	MBE=0.4368; MPE= -1.086; EF=0.9940
2.20	0.0017	2.073	14.65	MBE=1.104; MPE= -1.513; EF=0.9956
2.64	0.0018	2.115	13.85	MBE=0.9339; MPE= -2.318; EF=0.9954
4.40	0.0004	2.368	15.07	MBE=0.5589; MPE= -1.506; EF=0.9880
6.60	0.0003	2.476	14.69	MBE=0.8706; MPE= -3.040; EF=0.9781
11.0	0.0002	2.613	14.43	MBE=1.322; MPE= -4.184; EF=0.9694
13.2	0.0001	2.790	15.68	MBE=0.3920; MPE= -0.7708; EF=0.9661
22.0	0.0001	2.819	16.67	MBE=0.2491; MPE= -4.438; EF=0.9739

The model parameters, flow activation energies and apparent viscosity values in these theoretical models were determined by using statistical package program (Statistica for Windows 5.0) with non-linear regression analysis. Using some statistical tests, the reliability of the model equations was investigated and the consistency of the experimental apparent viscosity values and the apparent viscosity values calculated from the model were determined (Table 3 and Table 4).

Table 4. The change of agar solution to apparent viscosity with temperature and concentration (Eq. 12)

Shear rate (s^{-1})	$\eta_a= \delta_1 \exp(\varepsilon_1 C)\exp(E_a/RT)$			
	δ_1 (mPas)	ε_1 $(kg/m^3)^{-1}$	E_a (kJ/mol)	**Statistical tests**
1.32	0.8205	0.0698	11.89	MBE=2.559; MPE= -4.623; EF=0.9797
2.20	0.1795	0.0773	14.69	MBE=2.713; MPE= -5.835; EF=0.9862
2.64	0.2165	0.0787	13.90	MBE=3.075; MPE= -7.237; EF=0.9844
4.40	0.0863	0.0873	15.15	MBE=1.934; MPE= -7.384; EF=0.9861
6.60	0.0739	0.0909	14.76	MBE=1.923; MPE= -9.776; EF=0.9784
11.0	0.0544	0.0957	14.58	MBE=0.9383; MPE= -11.62; EF=0.9720
13.2	0.0250	0.1013	15.87	MBE=0.2519; MPE= -9.201; EF=0.9715
22.0	0.0144	0.1020	16.81	MBE=0.9957; MPE= -15.55; EF=0.9738

As can be seen from the applied statistical tests, the best model for expressing the combined effect of temperature and concentration on the apparent viscosity of agar solutions is Eq. 11.

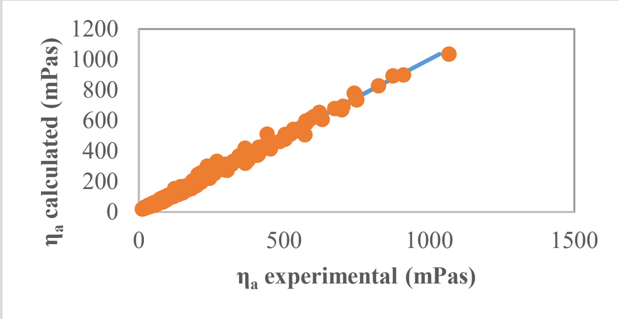

Figure 3. Comparison of the experimental apparent viscosity of agar solutions and the apparent viscosity values calculated from Eq. 11

The relationship between the experimental apparent viscosity values and the apparent viscosity values calculated from Eq. 11 is shown in Figure 3 for all shear rates, temperature and concentration.

The combined effect of temperature and concentration on viscosity is not simply additive or linear. The interaction between temperature and concentration can lead to complex and nonlinear changes in viscosity. The decrease in viscosity with increasing temperature was more pronounced at higher concentrations compared to lower concentrations [25].

Conclusions

As a result of the study, it was found that agar solutions exhibit a shear thinning (pseudo plastic) behavior due to the flow behavior index being less than 1. As a rheological model, it has been shown that it will be well represented by the power-law model. The viscosity of agar solutions increased with concentration. The increased viscosity of agar solutions with higher concentrations can be attributed to the larger number of agar molecules present in the solution. The viscosity of agar solutions decreased with an increase in temperature. Higher concentrations of agar, even with a decreasing viscosity trend with temperature, resulted in higher viscosities compared to lower concentrations at the same temperature. As a result of the study of the flow activation energy, it was found that the apparent viscosity of the agar solutions was relatively more sensitive to temperature change at high shear rates. The data obtained in the design of equipment necessary for processing agar solutions in the range of temperatures, concentrations and shear rates studied are valid.

Conflicts of interest

There are no conflicts of interest in this work.

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