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## On the Effects of Cold Temperatures and Solute Concentrations on the Solidification of Gelatine

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# On the Effects of Cold Temperatures and Solute Concentrations on the Solidification of Gelatine

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**Abstract.** The solidification phenomenon of gelatine is important in many applications especially as an ingredient in certain food products that requires particular textural characteristics. This work was aimed to study the effects of temperatures and solute concentrations on solidification kinetics of gelatine by using Avrami kinetic phase change model analyses. The extent of solidification/gelation was measured gravimetrically and the sigmoid solidification curves were able to be constructed. The Avrami plots were managed to be produced and the Avrami exponents,  $n$ , and the rate constant,  $K$ , were extracted from the linear equations obtained. It was found that, lowering the cooling temperatures lead to higher  $K$  and reduced  $n$  values depicting faster rate of solidification with smaller dimensions. It was also observed that increasing the solute concentration lead to larger  $K$  and lower  $n$  values portraying that solidification rates was increased, and higher dimensions of solids were achieved.

## 1. Introduction

Gelatine is colorless, translucent and brittle solid substance. It is one of hydrocolloids that can be extracted from collagen in animal bones and skins. Gelatine is high molecular polypeptides obtained by thermal denaturation of collagen which is the common starting ingredient [1]. It has many unique functional properties that serve many applications in daily life especially in food industries. It is also widely used in pharmaceuticals and biomedical products such as in the production of soft or hard capsules for drugs. Gelatine is also used in non-food applications such as in photographic paper and cosmetics.

One of the unique functional properties of gelatin is gelling that can be solid, or liquid based on the temperature. When gelatine is heated, it will form liquid jelly and solidify if cooled. The situation is known as thermo reversible gel. This will cause gelatin to solidify during the cooling process that is important in certain food such as desserts [2]. Several factors can affect the solidification of gelatine such as temperature and gelatin concentrations. The time-function-solidification phenomena can be studied thoroughly by analyzing the kinetics of solidification using Avrami phase change theory [3,4]. Utilisation of Avrami kinetics have been studied by many. Some of the studies used NMR as a tool for measuring the extent of gelation or solidification [5]; others used DSC [6]. In this work, simple gravimetric method was used to measure the extent of gelation.



Explanation on the concept and derivation of Avrami equation can be found elsewhere [7]. Important and meaningful parameters that can be obtained from Avrami analyses are the Avrami exponent,  $n$  and the rate constant,  $K$  [8]. The exponent,  $n$ , depicts the dimension of the solid/crystals whereas the constant  $K$  illustrates the growth rate of the solids/crystals. The objectives of this study were to investigate the effects of different cooling temperatures and the effects of different gelatin concentrations on the solidification of the solution.

## 2. Materials and Methods

The materials used in this study were beef gelatine powder, distilled water, sugar and citric acid. The beef gelatine powder was purchased from market in Alor Setar, Kedah and sugar were purchased from Jeli market in Kelantan. The citric acid was obtained from Merck Inc. The apparatus used in this study were beakers, measuring cylinder, spatula, digital thermometer, glass rod, magnetic stirrer, digital weighing balance, hot plate and water circulating bath.

### 2.1. Preparation of Gelatine Solutions

The halal gelatine powder was bought from the local supermarket. The powder was dissolved in hot distilled water of 60°C for 15 minutes by stirring using a hot plate magnetic stirrer until completely dissolved. The solution produced was clear and transparent.

### 2.2 Solidification of Gelatine Solution with Different Cooling Temperatures

Gelatine solution of 100 g of weight was placed into three different beakers and was heated by using hot plate until 60°C and the solution was fully liquified. The solidification of gelatine was determined by gravimetric method via separation of solid and liquid parts. The weight of both the poured liquid part was recorded from time to time with an interval of 1 minute. The process was repeated until the whole solution was fully solidified. Different cooling temperatures tested were 5°C, 10°C and 15°C which were controlled using cooled water bath.

### 2.3 Solidification of Gelatine Solution with Different Gelatine Concentrations

The setting index of gelatine depends on the time required for the gelatine solution to fully solidify in temperature of 5°C. The end time was considered when no more solution was able to be poured. Three different ratios (w/w) of gelatine powder and distilled water were selected i.e. 1:10, 1:20 and 1:30. The solutions were heated to the temperature of 60°C and solidification process were conducted by cooling them to 5°C. The extent of solidification again was determined gravimetrically. The measurement was recorded with time intervals of 1 minutes until it was fully solidified.

### 2.4 Plotting the Solidification Curve

The solidification of gelatine solution was determined gravimetrically i.e. by separating the solid and liquid parts into two different beakers at each time interval. The solid gel solution was weighed by using a digital weighing scale. The extent of solidification  $X_t$ , was calculated as:

$$X (\%) = (X_t / (X_\infty + X_0)) \times 100\%$$

Where  $X_t$  is the total solid deposition at time,  $t$  (min),  $X_\infty$ , the total mass of gelatine deposition obtained from the deposition curves when the asymptotic condition had been achieved (g) and  $X_0$  is the initial mass of gelatin content in liquid (g). Data obtained was recorded and analyzed. The curve of solidification percentage (%) versus time was plotted and the sigmoid graph was obtained.

### 2.5 Avrami Kinetics Analysis

The solidification percentage,  $X$  (%) data that had been obtained from section 2.7, was then used to construct the Avrami plot by using the equation below:

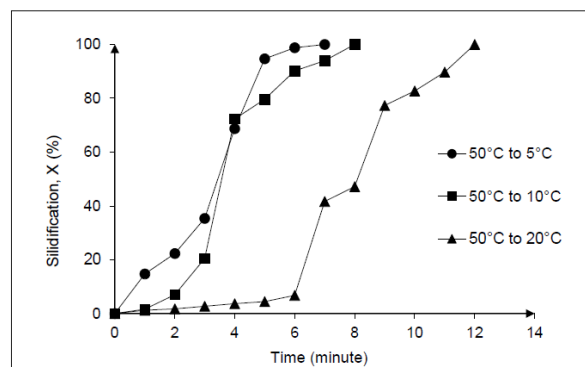
$$\log(-\ln(1-X)) = \log K + n \log(t)$$

A straight line plot of  $\log(-\ln(1-X))$  vs.  $\log(t)$  was plotted. This equation can be represented by a linear relation of  $Y = mX + C$ . Using the Excel functions, the trend line equation can be obtained and the Avrami exponent,  $n$  can be extracted from the slope value. Whereas the rate constant,  $K$  was obtained from the antilog of the constant  $C$  of the individual equation.

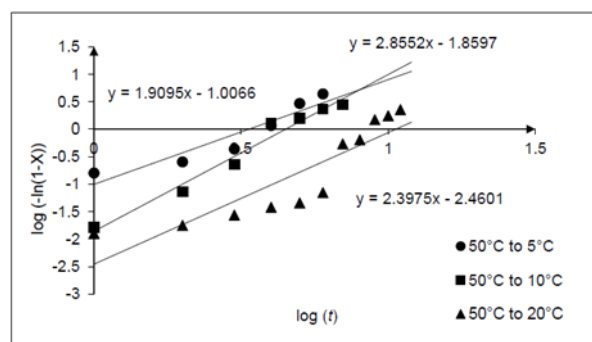
### 3. Results and Discussion

#### 3.1 Effects of Cold Temperatures

The gelatine solutions were cooled to 3 cold temperatures of 5°C, 10°C and 20°C. As shown in Fig. 1, the cold temperature of 5°C was having the fastest cooling rate, with the time taken to fully solidify the solution was around 7 minutes. As for the temperature of 10°C, full solidification time was 8 minutes, and for 20°C complete solidification was longest at 12 minutes. It was also can be observed that, the lower the cooling temperature, the higher the solidification percentage at any time. The data obtained then was manipulated to produce the Avrami plot as shown in Fig. 2. Straight line plots of  $\log(-\ln(1-X))$  versus  $\log(t)$  were produced and the  $n$  and  $K$  value were extracted and presented in Table 1.



**Figure 1.** Sigmoidal plots of effects of different cooling temperatures



**Figure 2.** Avrami plots for the effects of different cooling temperatures

From Table 1 it can be observed that the Avrami exponent,  $n$  values were increased when the cooling temperatures increased from 5°C to 10°C while decreasing again for the temperature of 20°C. The  $n$  values obtained was closed to 2 and 3 depicting that the dimension of the solids could be two or three dimensional in the form of plate or branchial spherulitic growth patterns. And from the  $K$  values, it can be deduced that increasing the cooling temperatures will decrease the rate constant. In

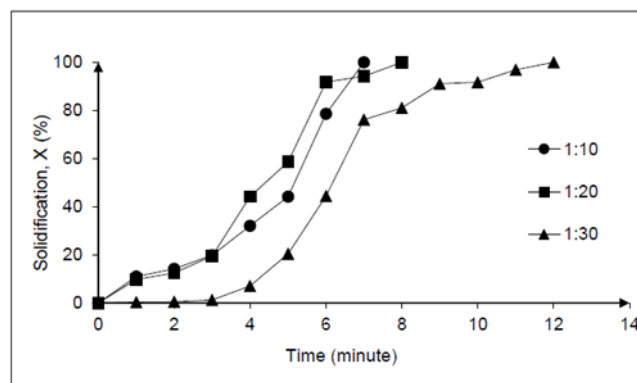
conclusion, reducing the cooling temperatures lead to faster production of the solids. It can also lead to the production of harder gelatine solids as flake type solids tends to be more rigid [7].

**Table 1.** Value of  $n$  and  $K$  extracted from the Avrami plots

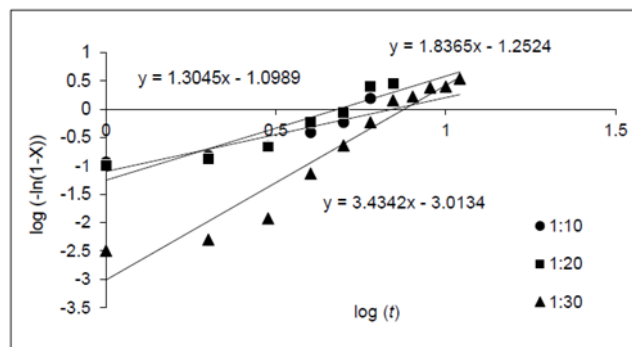
Cooling Temperature ( $^{\circ}\text{C}$ )	Heating Temperature ( $60^{\circ}\text{C}$ )	
	$n$	$K$ ( $\text{min}^{-1}$ )
5	1.91	0.099
10	2.86	0.014
20	2.40	0.004

### 3.2 Effects of Different Gelatine Concentrations

Three different concentrations were tested based on the ratio of gelatine to water which are 1:10, 1:20 and 1:30 (w/w). Solidification curves are shown in Fig. 3. As for the ratio 1:10, time required for gelatine solutions to fully solidified was 7 minutes whereas for the ratio of 1:20 took 8 minutes. Sample with the ratio of 1:30 solidified fully with the longest time of 12 minutes. It can be stated that, less concentrated solution required longer time for gelatine solution to solidify fully.



**Figure 3.** Effects of concentration on solidification



**Figure 4.** Avrami plots for the effects of concentrations

**Table 2.** Extracted Value of n and K for the effects of different concentrations.

Ratio of gelatine to water (Concentration)	Cold Temperature (5°C)	
	n	K (min <sup>-1</sup> )
1:10	1.30	0.080
1:20	1.84	0.006
1:30	3.43	0.001

From Table 2, it can be observed that the values of n and K was affected by different concentrations of gelatine. Increasing the concentrations, boost up the n values and decrease the K values. At higher concentrations, the growth rates of solidification were decreased meaning that the solutions will hardening faster. At higher concentration n values are lower, leading to the predictions that the solids produced could be of lower dimensions. This can produce harder gels due to more compact structures of the solids. Fig. 5 provide a visual representation of solid formation starting from the outside and moving inwards. The cloudy parts were the solids.

**Figure 5.** Photo showing solidification occurred from the outer surface inwards.

#### 4. Conclusion

It can be concluded that, lowering the cooling temperatures shorten the solidification time of gelatine solutions, reducing the dimensions of solids produced. Higher concentration of gelatine increased the solidification growth rates but the dimensions of the solids seemed to be increased.

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