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Comment on 'gelation mechanism of agarose and κ-carrageenan solutions estimated in terms of concentration fluctuation' [Polym 2002;43:5299]

In the paper by Matsuo and co-workers [1], differential scanning calorimetry (DSC) measurement was performed on agarose gel using an Exstar 6000 of Seiko Instruments Incorporation with a heating rate of 1 °C mm^{-1}. The weight of the sample was 38 mg. It was scanned over a temperature range of 0–80 °C during heating and 80 to ~12 °C during cooling. The DSC curves for agarose solution with 2.5% concentration both under heating and cooling show no peak. Accordingly, they concluded that crystallites in the gel were not present due to absence of endotherm peak. Of course, no exotherm peak suggests any chain arrangement under melting process of gel.

It is well known that gel formation is a multistep one exhibiting hysteresis and leading to variety of final structures depending on the particular solvent, i.e. water and environmental conditions [2–7]. In fact, gelation is often an outcome of different and mutually interacting processes, such as a conformational change, molecular cross-linking, and liquid–liquid phase separation [8–11]. The gelation mechanism in agarose depends primarily on the quenching temperature and polymer concentration [5]. It is also reported that at agarose concentration, larger than 1 wt%, molecular cross-linking dominates over spinodal demixing [12]. Cross-linking points in the gels are crystallites and take clear boundary surfaces [13]. In the light of the above it may be concluded that the DSC curves as observed by Matsuo and co-workers [1] suffer from serious flaws. In DSC setup there is a small crucible in which a small amount of sample can be put. The exposed sample area is large as compared to its volume. Due to slow heating the water molecules leave voids and interstitials of agar gel quite regularly and gradually. In this process the gel lost the water molecules. Finally, it reaches a state, where it can't be melt but burnt. An investigation was carried out on the agarose sample (2.5 wt%) using a scanning electron microscope (SEM) of FEI company (model: Quanta 200FEG) to see any morphological changes in agarose during heating. The mode heating in SEM is similar to that of DSC. Fig. 1(a) and (b), the SEM micrographs show no morphological changes during heating when the temperature of the specimen was raised up to 80 °C. It proves that no melting takes place even at a very high temperature. Thus, DSC study can't provide any information on gelation of agar.

The authors would like to state that an instrument to study the temperature dependent properties of agarose like hydrogels is to be equipped with an indirect heating arrangement to ensure a minimum change in water content. Moreover, when bulk heating is ensured by reducing surface area as compared with its total volume, any measurement of temperature dependent properties of agarose like hydrogels will be less erroneous. In conclusion, an indirect heating technique like microwave heating to be adopted in an instrument is suggested to have correct information on physical changes of agarose-like hydrogels with temperature.

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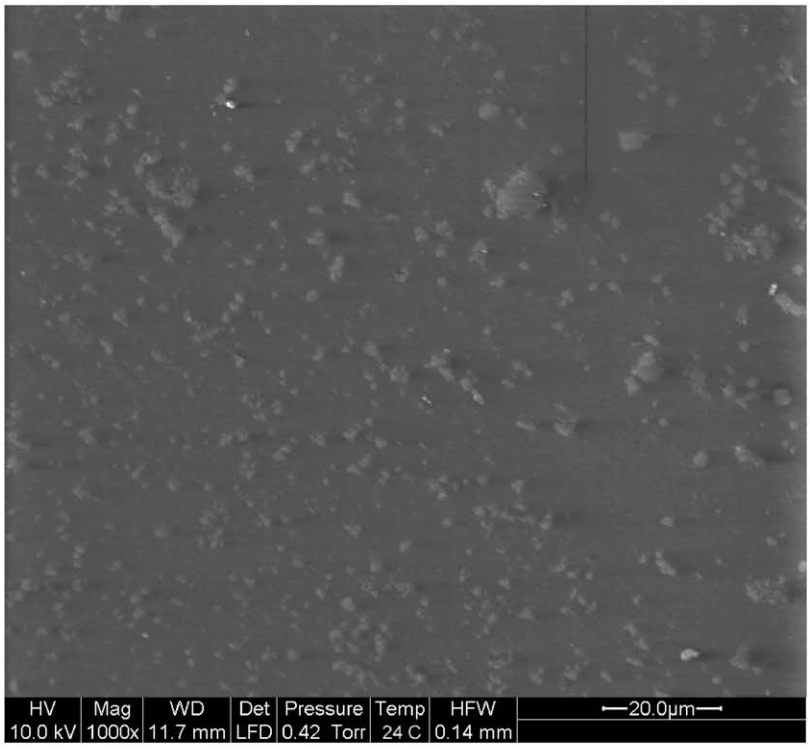
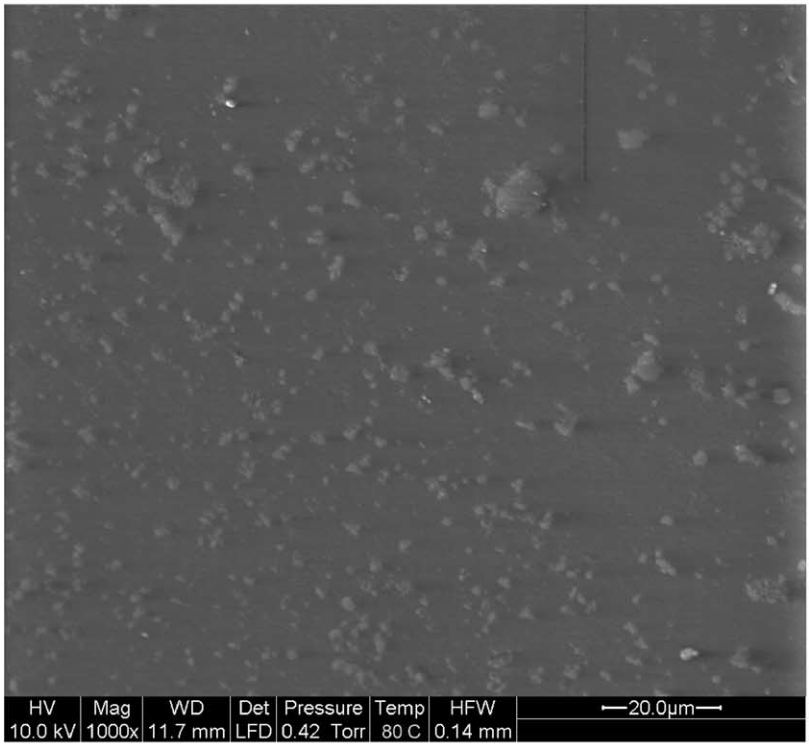


(a)



(b)

Fig. 1. SEM micrographs of agarose at (a) 24 °C and (b) 80 °C.

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