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# Steady, dynamic, and creep-recovery rheological properties of myofibrillar protein from grass carp muscle

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A B S T R A C T

This study investigated the solution behaviors and sol–gel transition of myofibrillar protein (MP) from grass carp muscle by rheological methods. In steady shear measurements, shear-thinning behavior was observed in the MP solutions and became more apparent with increasing concentration ($c$), though Newtonian plateaus appeared at $c \leq 0.025$ mg/mL. Based on the Cross model, zero shear viscosity ($\eta_0$) and structural relaxation time ($K$) increased with increasing $c$, indicating the reinforcement of chain interaction and entanglement. From the dependence of $\eta_0$ on $c$, the MP showed the rod-like polymer features due to its $\alpha$-helical tail, and the coil overlap concentration ($c^*$) was estimated to be 4.5 mg/mL, at which the MP coils begin to overlap and interpenetrate undergoing the transition from a dilute to concentrated solution. In frequency sweep, the MP system was classified to two regions: an entanglement network or concentrated solution (5 and 10 mg/mL), and a weak gel (15 and 20 mg/mL). Furthermore, the sol–gel transition concentration ($c_{gel}$) was further identified as 13 mg/mL by the Winter–Chambon criterion. According to the creep-recovery data fitted by the Burger model, the elastic coefficients ($G_1$ and $G_2$) and viscous coefficients ($\eta_1$ and $\eta_2$) increased with increasing temperature (5–40 °C), while the maximum deformation ($J_{MAX}$) and residual deformation ($J_\infty$) decreased, leading to an increase in the final percentage recovery ($R\%$). This result indicated that the chain rigidity of MP increased with elevated temperature, the MP gel network formed and became stronger, confirming the denaturation, aggregation and pre-gelation of MP at ~40 °C.



## 1. Introduction

The aquatic products processing industry is becoming one of three pillar industries with fishing and aquaculture in China's fish culture, and fish production predominates among the aquatic products (Li, Lu, Zhu, Wang, & Li, 2009). The surimi made of grass carp (*Ctenopharyngodon idellus*) is a kind of popular and commercial freshwater fish surimi in China because of its good flavor and mouthfeel associated with texture (Ding et al., 2012). Due to its rheological properties, myofibrillar protein (MP), the major constituent of salt-soluble proteins from grass carp muscle that can only be solubilized in high ionic strength, plays an essential role in the texture and processing characteristics of fish surimi products (Pérez-Juan, Flores, & Toldrá, 2007).

Rheological characterization is an extensively used and convenient strategy for investigating shear response of hydrogels, which are widely used in the food and pharmaceutical industries and classified by the type of crosslinking, i.e., chemically (covalently) crosslinked or physically crosslinked (based on secondary interactions such as hydrogen bonding, hydrophobic and electrostatic interactions) (Sathaye et al., 2015). Rheological behaviors not only have great influences on the manufacture, storage, as well as texture of hydrogels composed of proteins and polysaccharides, but also inherently reflect the molecular structure and chain conformation of macromolecules (Oliveira et al., 2011; Tan, Pan, & Pan, 2008; Xu, Xu, Zhang, & Zhang, 2008). Xu, Chen, and Zhang (2007) found that *Aeromonas* gum had high viscosity by means of steady shear tests, but it could not form gel even at high concentration,

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**Nomenclature**

| | |
|---|---|
| MP | myofibrillar protein |
| $c$ | concentration (mg/mL) |
| $\dot{\gamma}$ | shear rate ($s^{-1}$) |
| $\eta$ | apparent viscosity (Pa s) |
| $\eta_0$ | zero shear viscosity (Pa s) |
| $\eta_\infty$ | infinite shear viscosity (Pa s) |
| $K$ | Cross model constant ($s^n$) |
| $n$ | Cross model exponent (dimensionless) |
| $c^*$ | coil overlap concentration (mg/mL) |
| $G'$ | storage modulus (Pa) |
| $G''$ | loss modulus (Pa) |
| $\omega$ | angular frequency (rad/s) |
| $tan\delta$ | loss tangent (dimensionless) |
| $n$ | relaxation exponent of Winter–Chambon (dimensionless) |
| $J$ | compliance ($Pa^{-1}$) |
| $G_1$ | instantaneous elastic modulus (Pa) |
| $G_2$ | retarded elastic modulus (Pa) |
| $\eta_1$ | residual viscosity (Pa s) |
| $\eta_2$ | internal viscosity (Pa s) |
| $R^2$ | correlation coefficient (dimensionless) |
| $B$ | constant of recovery speed ($s^{-C}$) |
| $C$ | exponent of recovery speed (dimensionless) |
| $J_{element}$ | corresponding compliance parameters, $J_{SM}$, $J_{KV}$ or $J_\infty$ |
| $J_{MAX}$ | maximum deformation ($Pa^{-1}$) |
| $J_{SM}$ | instantaneous elastic compliance ($Pa^{-1}$) |
| $J_{KV}$ | delayed elastic compliance ($Pa^{-1}$) |
| $J_\infty$ | irreversible compliance ($Pa^{-1}$) |
| $R$ | final percentage recovery (%) |

due to its nature of a random coil-like polysaccharide. Zhang, Xu, and Zhang (2008) detected from dynamic oscillation measurements that lentinan could form gel network at low concentration and temperature, which was closely related to its triple-helical conformation. Additionally, creep-recovery experiments have been recommended to assess the most likely internal structure of a system, as well as its structural changes influenced by its composition (Dolz, Hernández, & Delegido, 2008). Ice cream and its composite with xanthan were studied by creep-recovery tests, and it was found that the increase in processing temperature caused an increase in compliance, indicating that the elastic bonds were broken irreversibly and the structure was weakened with higher chain flexibility. Meanwhile, xanthan could enhance the internal structure of ice cream as indicated by the elastic and viscous coefficients (Dogan, Kayacier, Toker, Yilmaz, & Karaman, 2013; Toker et al., 2013).

With respect to fish muscle products, several previous studies have reported their rheological characteristics (Jafarpour & Gorczyca, 2009; Kim, Kim, Gunasekaran, Park, & Yoon, 2013; Tahergorabi & Jaczynski, 2012; Yoon & Park, 2011). For instance, Jafarpour and Gorczyca (2009) compared the sol–gel thermographs of three species surimi of common carp, threadfin bream and Alaska pollock by temperature sweep test, and found the sol–gel transition was completed at about 69, 61 and 53 °C for common carp, threadfin bream and Alaska pollock surimi, respectively. Kim et al. (2013) investigated the concentration dependence of the mechanical modulus of xanthan–locust bean mixture and Alaska pollock surimi via frequency sweep tests to approximately estimate critical concentration based on the theories of universality and rubber elasticity. Actually, the rheological characteristics of fish muscle (or surimi) are governed by the myofibrillar protein because of its ability to form three dimensional gels upon heating and subsequent cooling (Verbeken, Neirinck, Van Der Meeren, & Dewettinck, 2005). Liu, Zhao, Xiong, Qiu, and Xie (2008) explored the effects of various concentrations of myofibrillar protein, ionic strength, temperature and pH on steady shear response. Liu, Bao, Xi, and Miao (2014) investigated the impact of pH on viscoelasticity of tuna myofibrillar protein in linear and nonlinear viscoelastic regions, and they found that myofibrillar protein had a stronger and stabler network at a higher pH (7.0) and the shear cycle required more energy to complete, but the mechanism underlying the rheological behavior and the relationship between the rheological behavior and structure remain unclear. Ding et al. (2012) found that fish myofibrillar protein could form gel and its gelation temperature exhibited strong concentration dependence by temperature sweep analysis. From the aforementioned reports, it can be seen that the sol–gel transition temperature or concentration was just roughly predicted by temperature sweep test or modulus-concentration relationship, but the systemic and in-depth studies by different rheological methods about the rheological behaviors of fish MP, especially the rheological information associated with the macromolecular structure, entanglement state and chain flexibility have not been available yet. Therefore, a comprehensive and intensive understanding on the stable, dynamic and creep-recovery rheological behaviors of MP system in terms of sol–gel transition based on Winter–Chambon criterion and Burger model is crucial to deduce the change in internal structure entangled network and interaction force induced by heating and concentration, further to guide the processing and formulation of fish surimi products.

In the present work, the rheological properties of MP influenced by various concentrations and temperatures were analyzed systematically by steady shear, dynamic oscillatory and creep-recovery tests. In steady shear tests, the coil overlap concentration from dilute to concentrated solutions were estimated by the concentration dependence of zero-shear viscosity according to the Cross model. In dynamic oscillatory tests, the sol–gel transition was studied and the concentration at the critical gelation was determined by the Winter–Chambon criterion. In creep-recovery tests, the temperature dependency of viscoelastic property was analyzed by the Burger model. This work will provide fundamental insights about the rheological properties based on concentration and temperature to reflect the MP structure.

## 2. Material and methods

### 2.1. Preparation of myofibrillar protein (MP) from grass carp

Myofibrillar protein (MP) was extracted from grass carp as described in our previous work (Ding et al., 2012). Grass carp was purchased from Huazhong Agricultural University market in Wuhan, Hubei, China. Each of the fresh fish weighing ~1.5 kg was washed after removing its scale, head and organs. The white meat on the back was collected and minced, followed by rinsing the fish mince (100 g) three times with 400 mL of low salt buffer (0.05 M NaCl, 20 mM Tris–HCl, pH 7.5) to remove water-soluble proteins, and then centrifugation at 4000 r/min, 4 °C using a refrigerated centrifuge (Avanti J-26 XP, Beckman Coulter, CA, USA). After