

Direct preparation of gels from herbal medicinal plants by using a low toxicity liquid zwitterion

メタデータ	言語: eng 出版者: 公開日: 2020-04-03 キーワード (Ja): キーワード (En): 作成者: メールアドレス: 所属:
URL	https://doi.org/10.24517/00056095

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Supplementary Information

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Chiaki Kodo, Kosuke Kuroda, Keisuke Miyazaki, Hisai Ueda, Kazuaki Ninomiya, and Kenji Takahashi*

Materials

Dry powder licorice was purchased from Hidejuro Nakatani Pharmaceutical Co., Ltd. (Ishikawa, Japan) and used as received. Standard glycyrrhetic acid, anhydrous sodium dihydrogen phosphate, and ultrapure water were purchased from Wako Pure Chemical Industries, Ltd. (Osaka, Japan) and used as received. Avicel® PH-101 as microcrystalline cellulose (MCC) was purchased from Sigma-Aldrich Co., LLC. (Tokyo, Japan) and used as received. Methanol, acetic acid, and disodium hydrogen phosphate were purchased from Nacalai Tesque Inc. (Kyoto, Japan) and used as received. Tributylamine, tris (hydroxymethyl) aminomethane, hydrochloric acid, and tert-butyl alcohol were purchased from Tokyo Chemical Industry Co., Ltd. (Tokyo, Japan) and used as received. Ethanol was purchased from Kanto Chemical Co., Inc. (Tokyo, Japan) and used as received. OE₂imC₃C was synthesized as reported.¹

LC/MS measurement

The LCMS-8030 instrument consists of LC-30AD, SIL-30AC, CBM-20A, SPD-20A, and CTO-20A. An L-Column 2 ODS 2.1×150 nm, 3 μ m (Chemicals Evaluation and Research Institute, Tokyo, Japan) was used. The mobile phase (A, 10 mM tributylamine and 14 mM acetic acid in ultrapure water; B, 100 % methanol) was eluted at a flow rate of 0.3 mL/min, and the injection volume was 5 μ L. The linear gradient was applied as A:B = 100:0 at 0 min, A:B = 75:25 at 7.5 min, A:B = 40:60 at 11 min, A:B = 20:80 at 13 min, A:B = 0:100 at 15 min. A:B = 0:100 was maintained from 15 min to 19.5 min. Then the gradient was shifted to A:B = 100:0 at 20 min. The column was maintained at 40 °C. Glycyrrhizic acid was detected in the negative mode at 821.5 m/z. The interface voltage was -3.5 kV. The nebulizer gas flow rate was 2.0 mL/min, and the drying gas flow rate was 15 L/min. The DL temperature was 250 °C. The heat block temperature was 400 °C, and the CID gas was 230 kPa.

Observation of the structure of prepared gels by scanning electron microscopy

To observe the structure of prepared gels by scanning electron microscopy, dry licorice gel samples for scanning electron microscopy were prepared by solvent exchange and freeze-

drying as reported.² Gels with 0, 1, or 2 wt% MCC were prepared using ultrapure water as the poor solvent. The gels were maintained for one week, and the ultrapure water was exchanged daily. After one week, the ultrapure water was changed to ethanol, then to tert-butyl alcohol as follows: gels were changed from ethanol to 50% tert-butyl alcohol for 10 min, and then changed in a stepwise manner to 60%, 70%, 80%, 90%, and 100% tert-butyl alcohol every 10 min. Each gel was then freeze-dried for 1 h (FDU-2200, EYELA, Tokyo Rikakikai Co., Ltd., Tokyo, Japan). The dried gels were coated with Au/Pd alloy by sputtering, and the gels were imaged by scanning electron microscopy (JSM-6510LV, JEOL Ltd., Tokyo, Japan).

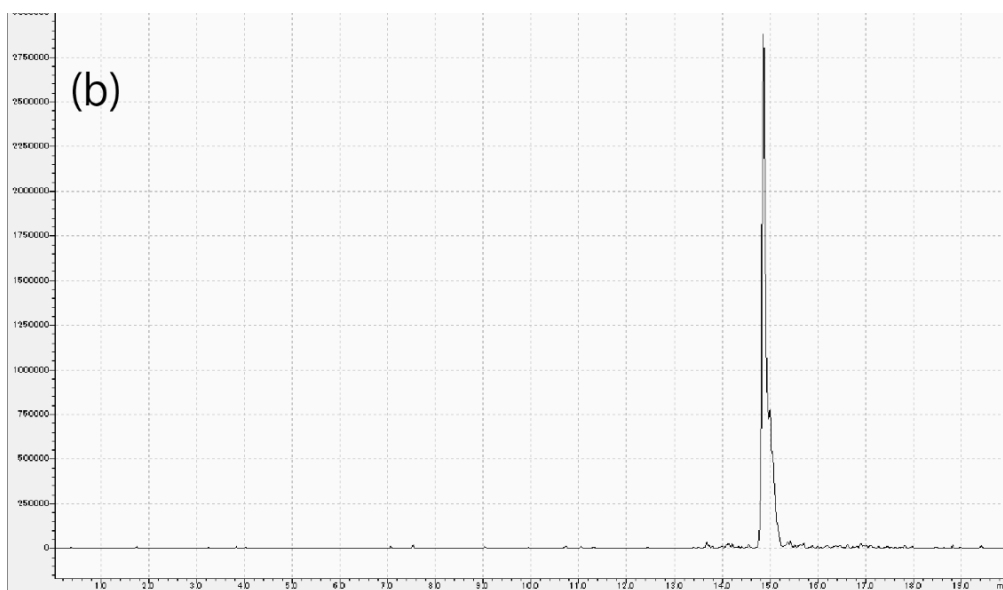
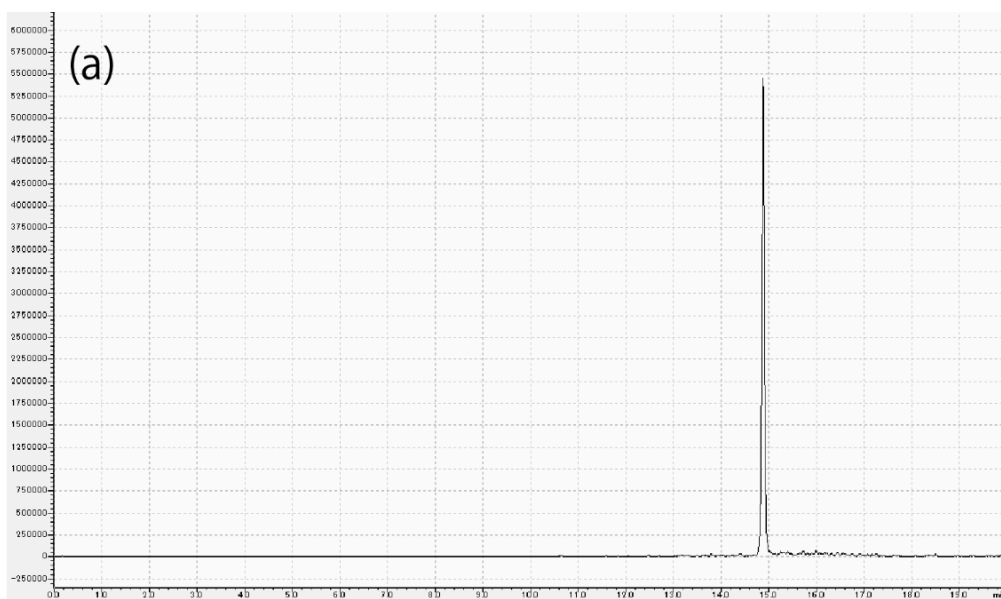


Fig. S1 Spectra of (a) glycyrrhizic acid solution and (b) a typical sample (corresponding to the sample at pH 7.4 and 24 h in Fig. 3).

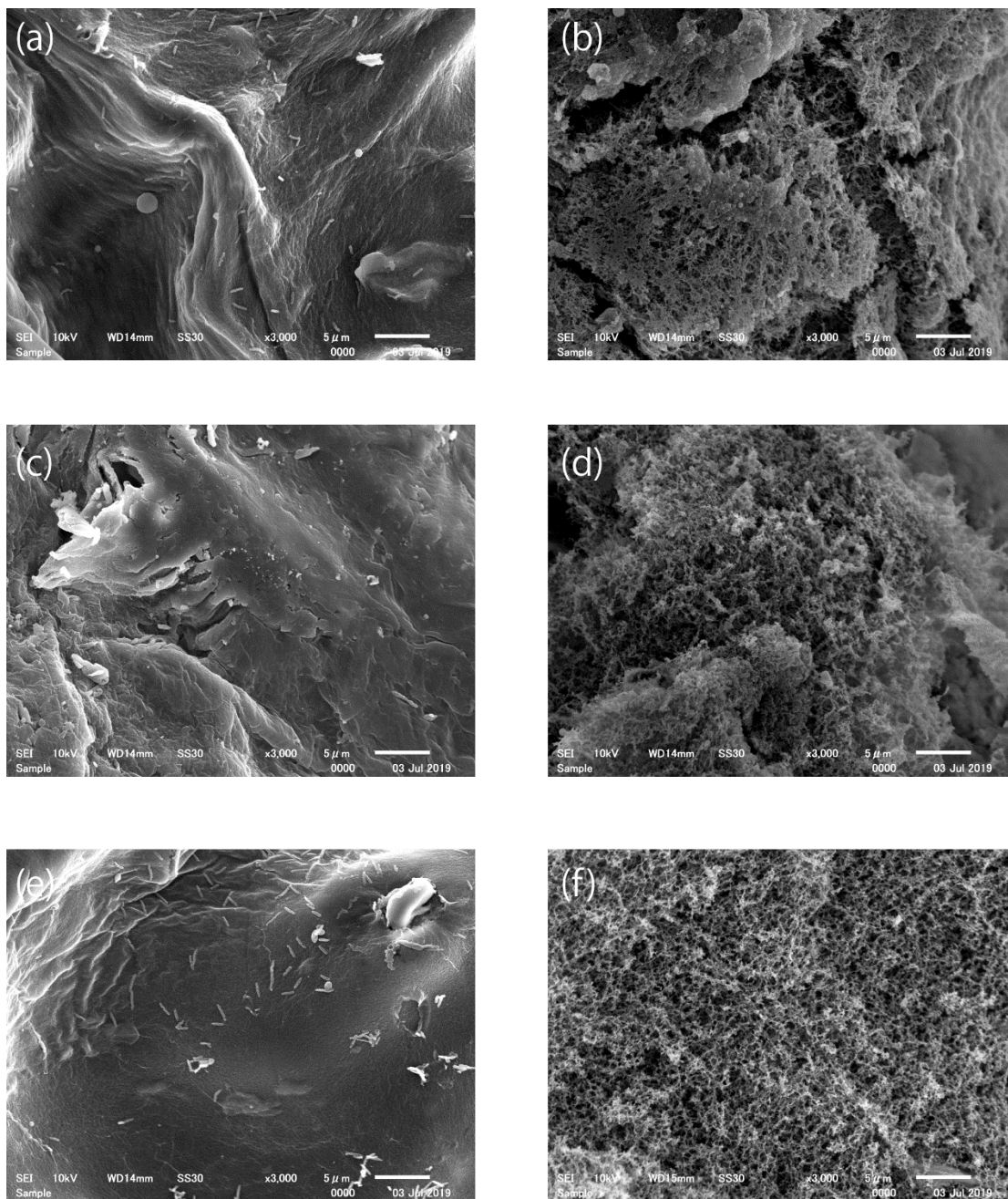


Fig. S2 The structures of (a) (c) (e) outer and (b) (d) (f) inner surfaces of the gels with (a) (b) 0 wt%, (c) (d) 1 wt%, (e) (f) 2 wt% of MCC, respectively.

References

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