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Cycling performance of lithium polymer cells assembled by in situ polymerization of a non-flammable ionic liquid monomer

Yoon-Sung Lee, Dong-Won Kim*

Department of Chemical Engineering, Hanyang University, Seungdong-Gu, Seoul 133-791, Republic of Korea

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ABSTRACT

Lithium polymer cells composed of a lithium negative electrode and a $LiCoO_2$ positive electrode were assembled with a gel polymer electrolyte obtained by in situ polymerization of an electrolyte solution containing an ionic liquid monomer with vinyl groups. The polymerization of the electrolyte solution containing the non-flammable ionic liquid monomer resulted in a significant reduction of the flammability of the gel polymer electrolytes. The lithium polymer cell assembled with the stable gel polymer electrolyte delivered a discharge capacity of 134.3 mAh g^{-1} at ambient temperature and exhibited good capacity retention.

1. Introduction

Rechargeable lithium batteries using lithium metal as a negative electrode are one of the most attractive candidates for high energy density power sources in portable electronic devices, electric vehicles and energy storage systems because lithium offers the largest specific capacity ($\sim$3860 mAh g^{-1}) as an active electrode material [1]. As an electrolyte system in lithium metal batteries, solid polymer electrolytes based on poly(ethylene oxide) (PEO) have been considered ideal alternatives to liquid electrolytes [2–5]. However, their low ionic conductivities, which range from 10^{-8} to 10^{-5} S cm^{-1}, preclude their practical application in lithium batteries at ambient temperatures. Gel polymer electrolytes consisting of a host polymer, an organic solvent and a lithium salt have been used to produce materials with high ionic conductivities and acceptable electrochemical stabilities [6–8]. Although the ionic conductivities of such gel polymer electrolytes usually exceed 10^{-3} S cm^{-1}, their mechanical strength is not good, which is one of the most critical deficiencies preventing their use in practical lithium batteries. In order to overcome this problem, in situ chemical cross-linking of liquid electrolyte has been reported [9–16] and employed in the production of commercial lithium-ion polymer batteries. In this process, an electrolyte solution containing cross-linking agents is injected into an already constructed cell and a chemical cross-linking reaction is carried out by heating the cell, which can resolve the leakage problem of organic solvents while maintaining good interfacial contact between the electrolyte and electrodes. Recently, ionic liquids have been considered promising safe electrolytes due to their unique properties such as a negligible vapor pressure, non-flammability, high oxidative stability and thermal stability [17–20]. Polymeric ionic liquids obtained by the polymerization of ionic liquid monomer have also attracted much attention as the host polymer in polymer electrolytes [21–28]. The main advantages of polymeric ionic liquid electrolyte-based electrolytes are less-flammability and exceptionally high anodic stability compared to conventional gel polymer electrolytes, which allows their use in safety-enhanced lithium batteries with high-voltage cathodes. Whereas extensive research has been devoted to polymeric ionic liquids, very few systems have been applied to lithium batteries due to their poor ionic conductivity at room temperature.

In this work, with the aim of developing highly conductive and less-flammable electrolytes based on a polymeric ionic liquid, a gel polymer electrolyte was synthesized by in situ polymerization of an ionic liquid monomer in a cell. An electrolyte solution containing a non-flammable ionic liquid monomer was injected into the cell composed of a lithium negative electrode and a $LiCoO_2$ positive electrode, and polymerization of the ionic liquid monomer was carried out by heating the cell. This process makes it possible to directly gelate the electrolyte solution in the cell and allows for the effective encapsulation of the organic solvents in the cell. In this paper, the ambient temperature cycling performances of lithium polymer cells assembled by in situ polymerization of an ionic liquid monomer are presented and compared with the performances of liquid electrolyte-based lithium batteries.

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* Corresponding author. Tel.: +82 2 2220 2337; fax: +82 2 2298 4101.
E-mail address: dongwonkim@hanyang.ac.kr (D.-W. Kim).

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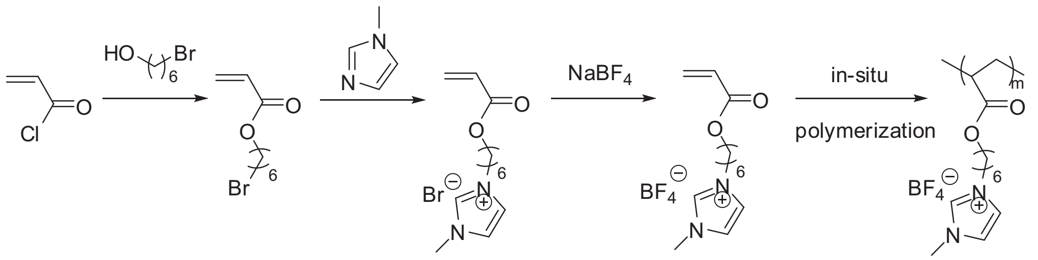


Fig. 1. Synthetic scheme of the polymeric ionic liquid, poly(1-methyl-3-(2-acryloyloxyhexyl) imidazolium tetrafluoroborate).

2. Experimental

2.1. Synthesis of ionic liquid monomer

The ionic liquid monomer was synthesized by the three-step process (Fig. 1) reported by Ohno's group [22,24]. 6-Bromo-1-hexanol (Aldrich) dissolved in tetrahydrofuran (THF) with an equimolar amount of triethylamine was fed into a glass reactor. A solution containing a slight molar excess of acryloyl chloride (Aldrich) in THF was added dropwise to the reactor over a period of 1 h under a N_2 atmosphere at 0 °C. After this solution was completely added, the resulting solution was vigorously stirred for 48 h. THF and residual monomers (6-bromo-1-hexanol, acryloyl chloride) were then removed in a rotary vacuum evaporator. Water and diethyl ether were added to the product, 1-boromohexyl-6-acrylate dissolved in diethyl ether was extracted from two separated layer, and diethyl ether was removed in a rotary vacuum evaporator. The obtained 1-boromohexyl-6-acrylate was reacted with n-methylimidazole in ethanol for 24 h at 45 °C. After evaporation of the ethanol, the product was washed twice with diethyl ether. The bromide anions in 1-boromohexyl-6-acrylate were replaced with tetrafluoroborate anions by applying sodium tetrafluoroborate in mixture of dichloromethane and de-ionized water using the conventional anion exchange method [29]. The ionic liquid monomer, 1-methyl-3-(2-acryloyloxyhexyl) imidazolium tetrafluoroborate (MAHI-BF_4), was vacuum dried at room temperature for 12 h.

2.2. Synthesis of gel polymer electrolytes

To prepare the gel electrolyte precursor, different amounts of ionic liquid monomer (5, 10, 15, 20 and 25 wt.%) were dissolved in the liquid electrolyte with t-amyl peroxypivalate (Luperox 554M75, Seki Arkema, 2 wt.% of ionic liquid monomer) as a thermal radical initiator. The liquid electrolyte used was 1.0 M $LiBF_4$ in ethylene carbonate (EC)/diethyl carbonate (DEC) (1:1 by volume, Soulbrain Co., Ltd., battery grade) containing 3 wt.% fluoroethylene carbonate (FEC). FEC was used as a solid electrolyte interphase (SEI) forming agent. The gel polymer electrolyte was then prepared by radical-initiated polymerization of the gel electrolyte precursor at 90 °C for 20 min in an Ar-filled glove box.

2.3. Electrode preparation and cell assembly

The positive electrode was prepared by coating the N-methyl pyrrolidone (NMP)-based slurry containing $LiCoO_2$ (Japan Chemical Co.), poly(vinylidenefluoride) (PVdF, KF-1100 Kureha) and super-P carbon (MMM Co.) (85:7.5:7.5 by weight) on aluminum foil. The electrode thicknesses ranged from 50 to 60 μm after roll pressing and the active mass loading corresponded to a capacity of about 1.2 mAh cm^{-2}. The negative electrode consisted of 100 μm thick lithium foil that was pressed onto a copper current collector. The lithium polymer cell was assembled by sandwiching the polypropylene separator (Celgard 3501) between the lithium electrode and $LiCoO_2$ electrode. The cell was enclosed in a metalized plastic bag, injected with the gel electrolyte precursor, and then vacuum-sealed in a dry box filled with argon gas. After the cell assembly process, the cell was maintained at 90 °C for 20 min in order to induce the in situ polymerization of the gel electrolyte precursor in the cell. After thermal polymerization within the cell, the internal overpressure in the cell could not be observed.

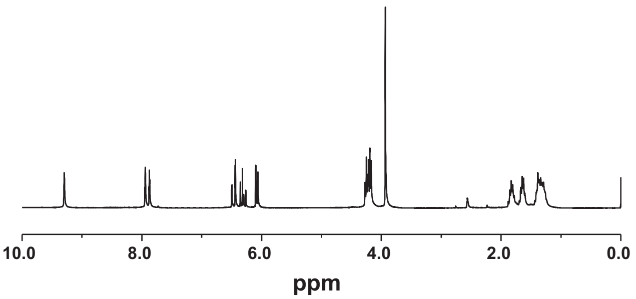
2.4. Measurements

The chemical structures of the ionic liquid monomer and polymeric ionic liquid were confirmed by ^1H NMR spectra using a VARIAN (Mercury 300) NMR spectrometer and a DMSO-d_6 solvent. The ionic conductivity of the liquid electrolyte was measured by a Cond 3210 conductivity meter (WTW GmbH, Germany) and the ionic conductivity of the gel polymer electrolyte was determined from AC impedance measurements. AC impedance measurements were performed using an impedance analyzer (Zahner Electrik IM6) over a frequency range of 100 Hz to 100 kHz with an amplitude of 10 mV. The self-extinguishing time (SET) was measured to quantify the flammability of the electrolyte, as previously described [30]. Briefly, the SET value was obtained by igniting the pre-weighed electrolytes, followed by measuring the time required for the flame to be extinguished. The electrolyte was judged to be non-flammable if the electrolyte did not ignite during the testing or if the ignition of the electrolyte ceased when the flame source was removed [31,32]. Charge and discharge cycling tests of the lithium polymer cells were conducted over a voltage range of 3.0–4.2 V at a constant current of 0.2 C and 25 °C using battery test equipment.

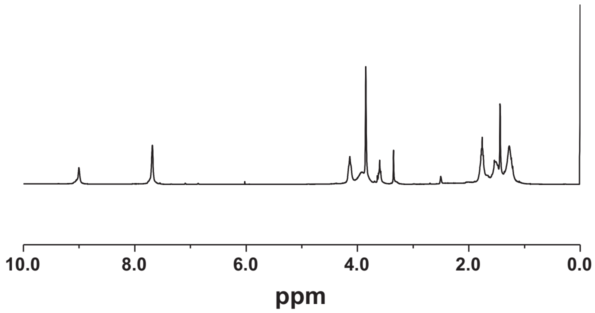
3. Results and discussion

The obtained ionic liquid monomer was transparent and pale yellow liquid at room temperature. Its melting point was measured to −27 °C from the differential scanning calorimetry experiments. The chemical structures of the ionic liquid monomer (MAHI-BF_4) and its polymeric product (P(MAHI-BF_4)) were confirmed by the ^1H NMR spectra shown in Fig. 2. The vinyl proton peaks ($CH_2{=}CH{-}$) in MAHI-BF_4 were observed in the range of 5.9–6.5 ppm, as shown in Fig. 2(a). After polymerization of the ionic liquid monomer, the peaks clearly disappeared and new proton peaks ($-CH_2-CH-$) corresponding to the polymer backbone appeared around 3.35 and 3.61 ppm, as shown in Fig. 2(b), indicating that the ionic liquid monomer is entirely polymerized to polymeric ionic liquid. The protons in the imidazole ring ($-CH_{imidazole}-$) appeared at 7.69 and 9.00 ppm. The methylene protons ($-CH_2-$) in side chain were observed in the ranges of 1.03–1.89 and 3.71–4.21 ppm. Thus, the ^1H NMR spectra of MAHI-BF_4 and P(MAHI-BF_4) are consistent with the expected chemical structures.

Before applying the gel electrolyte precursor into the cell, the gel polymer electrolyte was obtained by performing polymerization with different ionic liquid monomer contents in the liquid



(a) 1-methyl-3-(2-acryloyloxyhexyl) imidazolium tetrafluoroborate



(b) poly(1-methyl-3-(2-acryloyloxyhexyl) imidazolium tetrafluoroborate)

Fig. 2. ^{1}H NMR spectra of (a) 1-methyl-3-(2-acryloyloxyhexyl) imidazolium tetrafluoroborate and (b) poly(1-methyl-3-(2-acryloyloxyhexyl) imidazolium tetrafluoroborate) in DMSO-d_6.

electrolyte. Fig. 3 shows images of the gel polymer electrolytes obtained by polymerization with different amounts of the ionic liquid monomer. It is clearly seen that the electrolyte becomes non-fluidic and finally solidified when the content of the ionic liquid monomer increases, which is due to the polymerization of the ionic liquid monomer. A stable gel without flow was observed for the electrolyte system polymerized with an ionic liquid monomer content of 20 wt.%. This result suggests that the content of the ionic liquid monomer should be higher than 20 wt.% in the liquid electrolyte in order to induce effective gelation in the cell in order to prevent solvent leakage from the gel polymer electrolyte.

The ionic conductivities of the electrolyte systems before and after polymerization were measured as a function of the ionic liquid monomer content, as shown in Fig. 4. The ionic conductivity of the base liquid electrolyte was 8.3×10^{-3} S cm^{-1}. The ionic conductivities of the gel electrolyte precursor decreased with increasing ionic liquid monomer content since the addition of the ionic liquid monomer into the liquid electrolyte increases the viscosity of the resulting solution due to the increased ion-solvent and coulombic interactions between the ionic species. After polymerization of the gel electrolyte precursor, the ionic conductivities of the gel polymer electrolytes decreased compared to the gel electrolyte precursor with the same ionic liquid monomer content. It should be noted

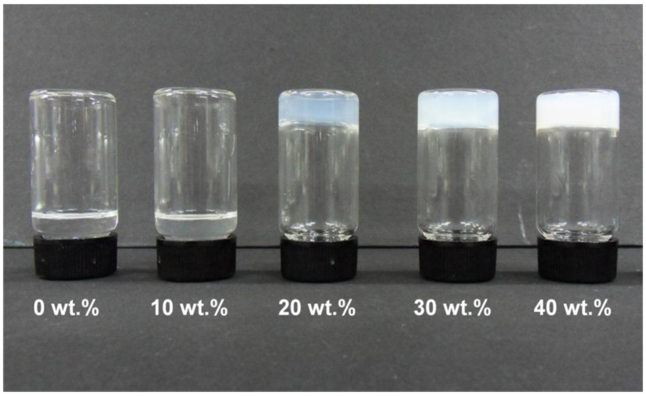


Fig. 3. Images of the gel polymer electrolytes obtained by polymerizing gel electrolyte precursors containing different amounts of the ionic liquid monomer.

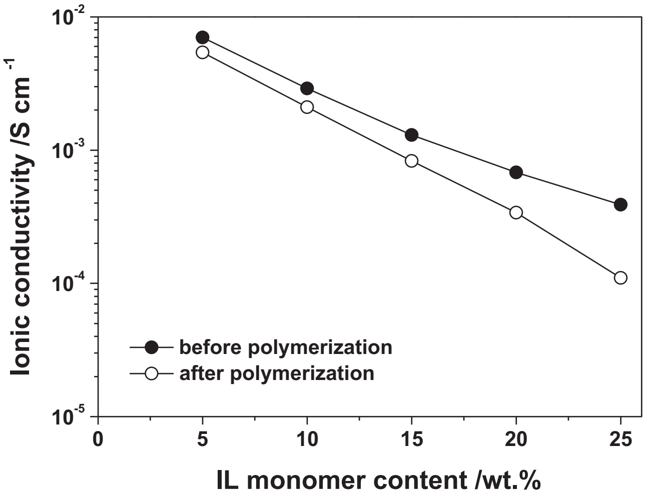


Fig. 4. Ionic conductivities of the electrolyte systems before and after polymerization of gel electrolyte precursors containing different amounts of the ionic liquid monomer at 25 °C.

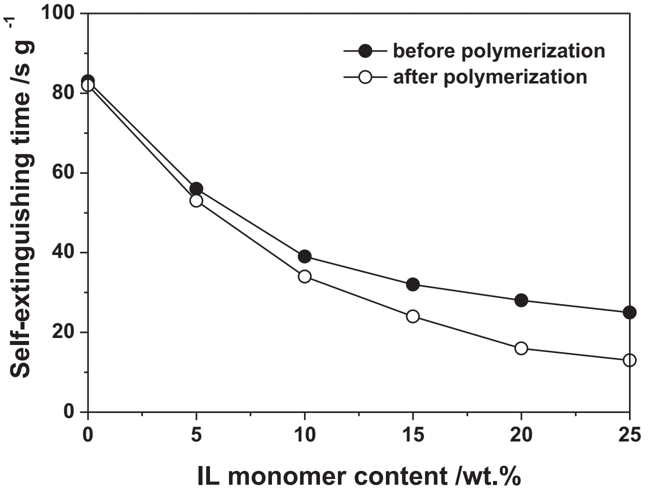


Fig. 5. SET values of the electrolyte systems before and after polymerization of gel electrolyte precursors containing different amounts of the ionic liquid monomer.

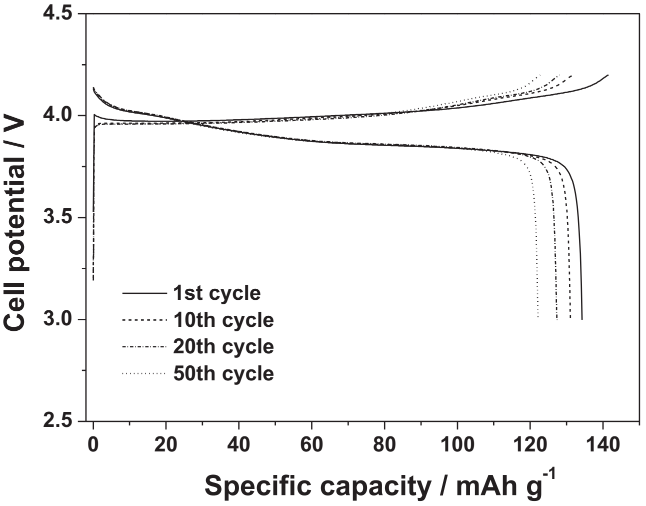


Fig. 6. Charge–discharge curves of a lithium polymer cell assembled with the gel polymer electrolyte obtained by polymerizing the gel electrolyte precursor containing 20 wt.% ionic liquid monomer. Cycling was carried out between 3.0 and 4.2 V at a current rate of 0.2 C at 25 °C.

that the reduction of the ionic conductivity after polymerization increased as the ionic liquid monomer content was increased. The large decrease of the ionic conductivity with increasing ionic liquid monomer content can be ascribed to the reduction of ionic mobility due to chain entanglement of the polymeric ionic liquid in the gel polymer electrolyte.

In order to compare the flammable behavior of the electrolyte systems containing different contents of ionic liquid monomer, flammability tests were performed before and after the polymerization. As reported earlier [30,33], the flammability of an electrolyte is proportional to the SET value. The SET values of the different electrolyte systems are shown in Fig. 5. Both the ionic liquid monomer and polymeric ionic liquid considered in this study did not exhibit any combustion, even during ignition with a flame source (i.e., their SET values are 0 s g^{-1}), indicating that all of them are non-flammable. When the ionic liquid monomer was added into the base liquid electrolyte, both the burning time and the flame intensity decreased, resulting in a reduced flammability. Accordingly, the SET values of the electrolyte systems decreased upon the addition of the ionic liquid monomer. As shown in Fig. 5, the SET values of the gel polymer electrolytes obtained after polymerization are lower than those of the gel electrolyte precursor. The reduction of flammability after polymerization may be related to the retarded leakage of flammable organic solvents from the gel polymer electrolytes. These results suggest that the addition of a non-flammable ionic liquid monomer to a base liquid electrolyte is effective in reducing the flammability of the gel polymer electrolyte obtained from its polymerization.

The cycling performance of the lithium polymer cells assembled by in situ polymerization was evaluated at 25 °C. Fig. 6 shows the charge–discharge curves of the 1st, 10th, 20th and 50th cycle of a lithium polymer cell assembled with the gel polymer electrolyte obtained by polymerizing the gel electrolyte precursor containing 20 wt.% ionic liquid monomer. The cell initially delivered a discharge capacity of 134.3 mAh g^{-1} with a coulombic efficiency of 94.9%, and the discharge capacity of the cell declined to 122.2 mAh g^{-1} after 50 cycles. The coulombic efficiency increased with cycling and remained greater than 99.0% throughout cycles after the initial few cycles. Without FEC as an additive, the coulombic efficiency of the cell with the gel polymer electrolyte synthesized from 20 wt.% ionic liquid monomer was low (92. 4%) at the first cycle and, it was lower than the that of cell containing FEC over the cycles. Low coulombic efficiency of the cell without FEC is associated with an irreversible reductive decomposition of polymeric ionic liquid, which indicates that FEC plays a role as a SEI forming agent for suppressing the reductive decomposition of electrolyte, as previously reported [20,34]. The stable SEI layer could not be formed in the ionic liquid monomer without FEC, though the pyrrolidinum-based ionic liquids had SEI forming ability on lithium electrode [35].

Fig. 7 compares the discharge capacity as a function of the cycle number in the cells containing the liquid electrolyte and gel polymer electrolytes. As seen in the figure, the initial discharge capacity of the cell decreased with increasing ionic liquid monomer content in the gel electrolyte precursor. As mentioned earlier, the polymerization of the gel electrolyte precursor with a high ionic liquid monomer content causes an increase of the resistance to ion migration in both the electrolyte and the $LiCoO_2$ electrode, which results in an increase of the internal cell resistance and a decrease of the discharge capacity. With respect to the capacity retention, there was little effect of the ionic liquid monomer content on the cycling stability of the cell. Based on these results, it is expected that gel polymer electrolytes obtained from the in situ polymerization of an ionic liquid monomer are promising, provided that the

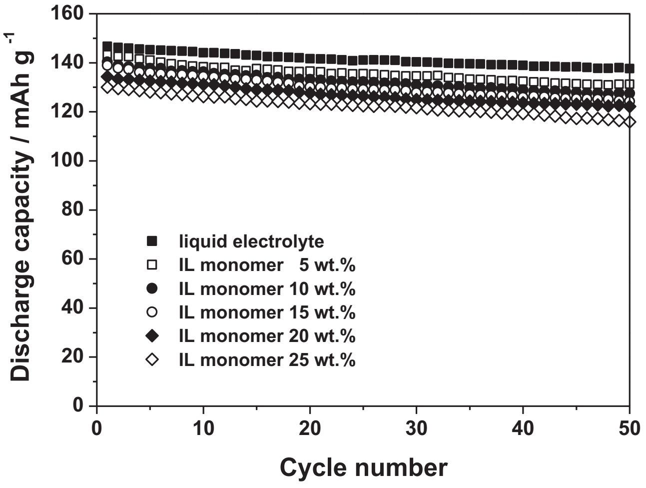


Fig. 7. Discharge capacities as a function of the cycle number of cells assembled with liquid electrolyte and gel polymer electrolytes. Cycling was carried out between 3.0 and 4.2 V at a current rate of 0.2 C at 25 °C.

ionic conductivity of the gel polymer electrolyte can be increased to result in lithium polymer cells with high capacities. More studies focusing on polymeric ionic liquid-based gel polymer electrolytes which do not result in a significant reduction of the ionic conductivity after in situ polymerization are currently in progress.

4. Conclusions

Gel polymer electrolytes based on a non-flammable polymeric ionic liquid were prepared and employed as the electrolyte material in lithium polymer cells composed of a lithium negative electrode and a $LiCoO_2$ positive electrode. The ionic liquid monomer content required to ensure stable polymer gel without leakage of solvent was demonstrated to be about 20 wt.%. The flammability of the electrolyte was significantly reduced by polymerizing the gel electrolyte precursor containing a non-flammable ionic liquid monomer. The in situ polymerization of the gel electrolyte precursor containing ionic liquid monomer decreased the ionic conductivity of the electrolyte, resulting in a decreasing discharge capacity with increasing ionic liquid monomer content. The cell assembled with the gel polymer electrolyte polymerized by the 20 wt.% ionic liquid monomer delivered an initial discharge capacity of 134.3 mAh g^{-1} at room temperature and exhibited good capacity retention.

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