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Effects of the LiFePO_4 content and the preparation method on the properties of $(\text{LiFePO}_4+\text{AC})/\text{Li}_4\text{Ti}_5\text{O}_{12}$ hybrid battery–capacitors

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Abstract: Two composite cathode materials containing LiFePO_4 and activated carbon (AC) were synthesized by an *in-situ* method and a direct mixing technique, which are abbreviated as LAC and DMLAC, respectively. Hybrid battery–capacitors $\text{LAC}/\text{Li}_4\text{Ti}_5\text{O}_{12}$ and $\text{DMLAC}/\text{Li}_4\text{Ti}_5\text{O}_{12}$ were then assembled. The effects of the content of LiFePO_4 and the preparation method on the cyclic voltammograms, the rate of charge–discharge and the cycle performance of the hybrid battery–capacitors were investigated. The results showed the overall electrochemical performance of the hybrid battery–capacitors was the best when the content of LiFePO_4 in the composite cathode materials was in the range from 11.8 to 28.5 wt. %, while the preparation method had almost no impact on the electrochemical performance of the composite cathodes and hybrid battery–capacitors. Moreover, the hybrid battery–capacitor devices had a good cycle life performance at high rates. After 1000 cycles, the capacity loss of the $\text{DMLAC}/\text{Li}_4\text{Ti}_5\text{O}_{12}$ hybrid battery–capacitor device at 4C was no more than 4.8 %. Moreover, the capacity loss would be no more than 9.6 % after 2000 cycles at 8C.

Keywords: hybrid battery–capacitor; LiFePO_4 content; preparation method; long cycle life; high rate; electrochemical performance.

INTRODUCTION

The emergence of hybrid electric vehicles (HEVs) requires the development of power sources that can provide high power as well as high energy.^{1–4} However, neither secondary batteries (low power density) nor capacitors (low energy density) as a single power source for HEVs can meet the needs of practical application.^{5–7} A hybrid battery–capacitor is a new energy storage system in which

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the secondary battery and capacitor coexist. It consists of an oxidation–reduction anode and a composite cathode composed of an oxidation–reduction battery electrode and an electric double-layer capacitor carbon electrode. During the charge–discharge process, the two oxidation–reduction electrodes form a battery system of oxidation–reduction cathode/oxidation–reduction anode, while at the same time, the electric double-layer carbon electrode and the oxidation–reduction anode form a hybrid capacitor system; hence the energy storage of the electrodes have not only Faradic energy but also electric double-layer energy. Therefore, a battery system and a capacitor system coexist in such an energy storage device, each of which brings its own good electrochemical performance into the whole system. The hybrid battery–capacitor has an energy density as high as the secondary battery and a power density as high as the capacitor.^{8–11} This makes it one of the most promising auxiliary power supplies for HEVs. Previously, a composite cathode material containing LiFePO₄ and activated carbon (AC), abbreviated as LAC, was synthesized by an *in-situ* method. Although the assembled hybrid battery–capacitor LAC/Li₄Ti₅O₁₂ showed the advantages of a high rate capability and high capacity,⁸ the *in-situ* synthesis required high system stability. It is hard to control product performance, stability and consistency, particularly an accurate content of LiFePO₄ in composite materials. The shortage of *in-situ* methods of synthesis increased the difficulty of industrial production. One possible solution to this problem is to synthesize the products by a direct mixing technique. In a direct mixing technique, the composite materials are synthesized *via* high-speed mechanical mixing, which is simple and the content of each component in composite materials can be easily controlled.

In this study, through the direct mixing technique, a series of composite materials containing commercial LiFePO₄ and AC, abbreviated as DMLAC, with different LiFePO₄ contents was synthesized. The effects of the LiFePO₄ content and preparation method on the electrochemical performance of the hybrid battery–capacitors were systematically studied.

EXPERIMENTAL

The LAC composite material was prepared *via* the *in-situ* method described in the literature.⁸ Li₄Ti₅O₁₂ powder was prepared *via* a solid-state reaction.⁸ The DMLAC composite material was prepared *via* the direct mixing technique. A stoichiometric mixture of LiFePO₄ and AC was dispersed in distilled water (the solid content was about 30 or 35 mass %) and then stirred at 2000 rpm for 6 h to give the final LiFePO₄+AC (DMLAC) composite material.

X-Ray diffraction (XRD) analysis of the electrode materials was realized a Philips X'Pert automated X-ray diffraction machine with CoK α 1 radiation of $\lambda = 0.178897$ nm, 50 kV, 35 mA, in the range of $15^\circ < 2\theta < 85^\circ$. Scanning electron microscopy (SEM) was conducted on a JEOL JSM-6700F scanning electron microscopy at 5 kV. The tap-density of the powders was tested by adding a weighed amount powder into a dry measuring cylinder and then the measuring cylinder was tapped until the volume of the powders no longer changed. The ratio of the mass and the volume of the powder gave the tap-density.¹²

A well mixed slurry of the active material LAC powder (or DMLAC, Li₄Ti₅O₁₂, LiFePO₄, AC), the electric conducting agent acetylene black and an aqueous binder LA132 (from Indigo, China) in a weight ratio of 85:10.5:4.5 was pasted onto aluminum foil and dried at 120 °C to give the electrodes. All electrodes were cut into discs with a diameter of 1.0 cm (*i.e.*, an area of 0.785 cm²), pressed, dried at 90 °C under vacuum for 4 h, and then stored in an argon-filled dry box. The hybrid battery-capacitor LAC/Li₄Ti₅O₁₂ (or DMLAC/Li₄Ti₅O₁₂) was assembled in an argon-filled dry box by pressing an LAC (or DMLAC) disc, a Celgard 2400 polypropylene membrane and a Li₄Ti₅O₁₂ disc. The electrolyte was a 1.0 M LiPF₆ solution in the mixture of ethylene carbonate (EC), dimethyl carbonate (DMC) and ethylene methyl carbonate (EMC) at a weight ratio of 1:1:1. For comparison, the capacitors of AC/Li₄Ti₅O₁₂ and AC/AC and the battery LiFePO₄/Li₄Ti₅O₁₂ were assembled in the same way. The constant current charge-discharge, rate capability and cycle performance of the hybrid battery-capacitors, capacitors and battery were tested on a Qing-tian battery tester (BS9300, China) and cyclic voltammograms were recorded on an Arbin instrument (USA).

In order to show the cycle life at a high rate, two kinds of DMLAC/Li₄Ti₅O₁₂ hybrid battery-capacitor devices were assembled. The cathodes were prepared with LiFePO₄ (STL, China) and AC (GH-6, China). The conductive additives used were colloidal graphite (F-0, China) and conductive carbon black (SuperP, TIMCAL) termed SP hereinafter. The binder used was LA132 (Indigo, China). Anodes were prepared in the same way using Li₄Ti₅O₁₂ as the active material. The formulations of the prepared electrodes are summarized in Table I. The cells were assembled in an argon-filled dry box by pressing a DMLAC cathode, a Celgard 2400 polypropylene membrane, and a Li₄Ti₅O₁₂ anode. The electrolyte was a 1.0 M LiPF₆ solution in a mixture of EC, DMC and EMC (1:1:1 by weight). The cells were packaged with an aluminum-plastic membrane and shaped. The final size of hybrid battery-capacitor was 4.5 mm×30 mm×48 mm (thickness×width×length). The cycle life performance of the hybrid battery-capacitors were tested on a Repower Battery Tester (China). All the tests were performed at 20 °C.

TABLE I. Formulations of the tested electrodes

Electrode	Active material	Conductive additives	Binder
15 % LFP + AC	LiFePO ₄ 15 % + AC 60 %	F-0 12 % + SP 6 %	LA132 7 %
22.5 % LFP + AC	LiFePO ₄ 22.5 % + AC 52.5 %	F-0 12 % + SP 6 %	LA132 7 %
LTO	Li ₄ Ti ₅ O ₁₂ 86 %	F-0 6 % + SP 4 %	LA132 4 %

RESULTS AND DISCUSSION

Analyses of XRD data

The XRD patterns of the LAC composites with different LiFePO₄ contents from 5.1 to 34.2 wt. % are shown in Fig. 1. Except for characteristic patterns of a small amount of Graphite-2H and Li₄P₂O₇, all the peaks are in accordance with the reference LiFePO₄ pattern (PDF No. 40-1499). Graphite-2H is formed because AC can transform to graphite at high temperatures.^{13,14} Li₄P₂O₇ is derived from the decomposition of LiFePO₄ at high temperatures.¹⁵ The decomposition process may be represented by the following reaction:



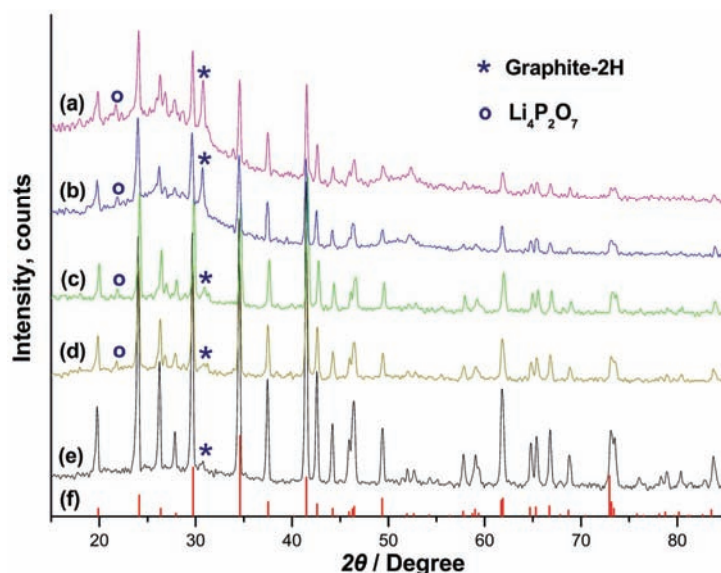


Fig. 1. XRD Patterns of the LAC composites with different LiFePO_4 contents: a) 5.1, b) 11.8, c) 21.1, d) 28.5, e) 34.2 and f) 100 wt. % (*i.e.*, the reference, LiFePO_4).

With increasing LiFePO_4 content, the XRD patterns became more similar to the reference pattern and the peak intensities of the impurities were correspondingly decreased.¹⁶ Moreover, the tap-density of the LAC composite increased with increasing LiFePO_4 content, which would improve the volume density of the composite cathode and the volume energy density of the hybrid battery–capacitor. The tap-densities of the LAC composites with different LiFePO_4 contents are listed in Table II.

TABLE II. The tap-density of the LAC composites with different LiFePO_4 contents

Content of LiFePO_4 , wt. %	0	5.1	11.8	21.1	28.5	34.2
Tap-density, g cm^{-3}	0.547	0.667	0.691	0.732	0.751	0.786

Morphologies of the samples

The SEM images of LAC composites with different LiFePO_4 contents are shown in Fig. 2. The images provide clear evidence that small crystal particles of LiFePO_4 either coat the surface of the AC particles or lie among them. These LiFePO_4 particles have a small particle size (100–500 nm) and a regular quadrangle form. With the increasing of LiFePO_4 content, these small crystal particles at the surface or among the AC particles are increased.

Cyclic voltammetric behavior

The cyclic voltammograms (CVs) of the hybrid battery–capacitors LAC/ $\text{Li}_4\text{Ti}_5\text{O}_{12}$ and DMLAC/ $\text{Li}_4\text{Ti}_5\text{O}_{12}$ are shown in Fig. 3. The CVs were all recorded

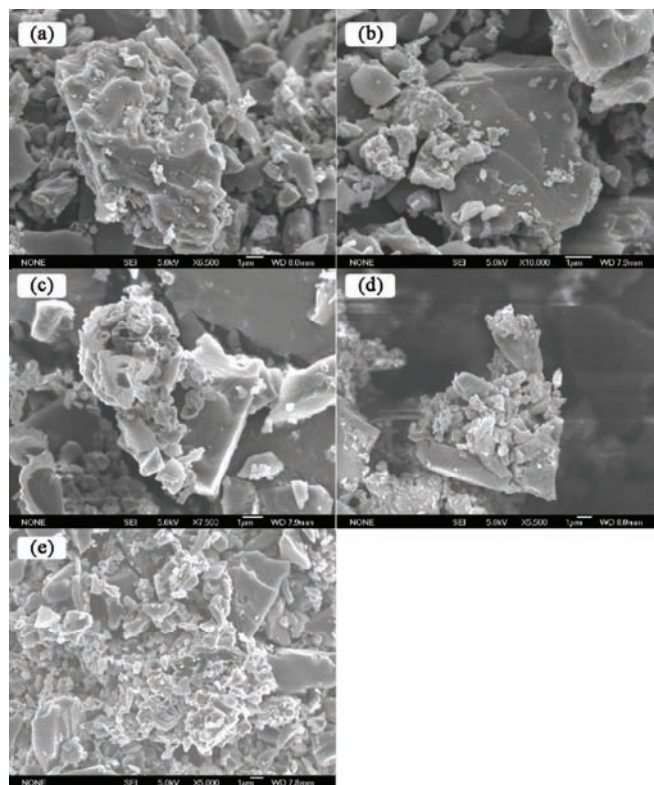


Fig. 2. SEM Microphotographs of the LAC composites with different LiFePO₄ contents: a) 5.1, b) 11.8, c) 21.1, d) 28.5 and e) 34.2 wt. %.

in the voltage range 1.0–2.6 V at a scan rate of 0.2 mV s⁻¹. As can be seen from Figs. 3a and 3b, the two hybrid battery–capacitors using the composite cathode prepared by the two methods exhibited the same electrochemical behavior. The hybrid battery–capacitor system retained the oxidation–reduction behavior of a LiFePO₄/Li₄Ti₅O₁₂ battery system. Furthermore, there was a larger response current when the hybrid battery–capacitor system was in the high voltage range. As shown in Figs. 3c and 3d, with a LiFePO₄ content of 11.8 wt. %, a pair of redox peaks of the LAC and DMLAC composites were both located at 1.74 and 2.00 V. With a LiFePO₄ content of 21.1 wt. %, the pair of redox peaks of the LAC composite was still located at 1.74 and 2.00 V. However, the pair of redox peaks of the DMLAC composite was located at 1.69 and 2.05 V. This result indicates that the preparation method of composite materials have a certain impact on the peak potentials of the CVs. The composite obtained by *in-situ* synthesis has small particles, a uniform distribution and no reunion phenomena. Therefore, the LiFePO₄ had a good interface combination with AC, a high degree of dispersion and weak electrode polarization, which is due to the good dispersion of LiFePO₄ in the LAC

composite. Moreover, the increase of the LiFePO_4 content did not affect their degree of dispersion in the LAC composite; hence the pair of redox peak was still located at 1.74 and 2.00 V. Compared with the direct mixing technique, LiFePO_4 in the DMLAC composite was not well-dispersed in the AC, had a weak interface combination with AC and a large electrode polarization. The increase in the LiFePO_4 content lowered the dispersion degree of LiFePO_4 in the DMLAC composite. Therefore, the pair of redox peak changes from 1.74 and 2.00 V (11.8 wt. % LiFePO_4) to 1.69 and 2.05 V (21.1 wt. % LiFePO_4). The above results of the CVs illustrate that the hybrid battery–capacitors LAC/ $\text{Li}_4\text{Ti}_5\text{O}_{12}$ and DMLAC/ $\text{Li}_4\text{Ti}_5\text{O}_{12}$ exhibited the same CVs behavior, but there was a difference in the redox peak potentials due to the different preparation methods.

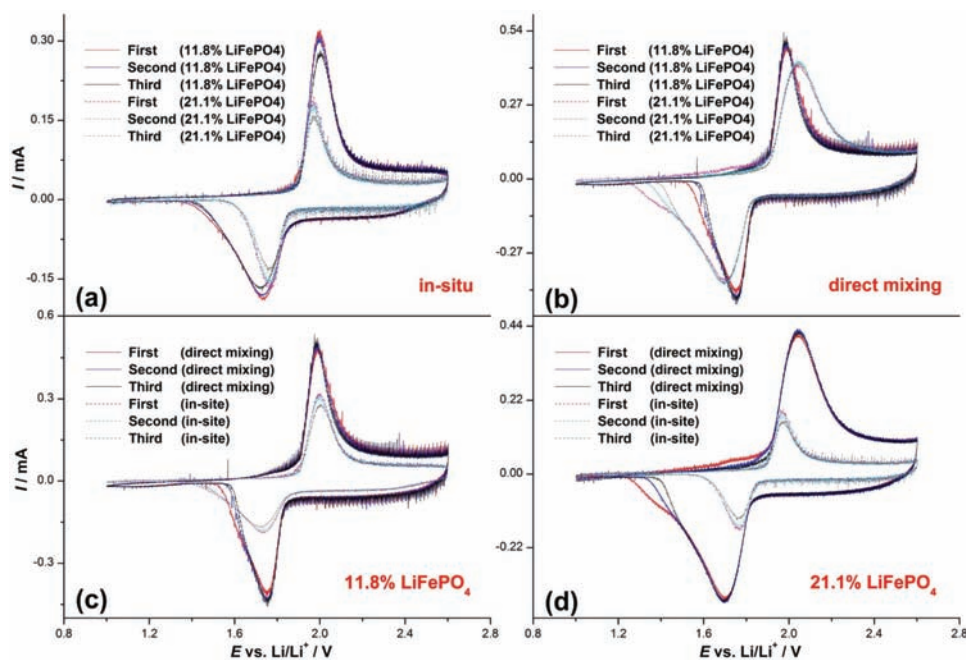


Fig. 3. CVs of the hybrid battery–capacitors with different LiFePO_4 contents: a) *in-situ* synthesis, b) direct mixing technique, c) 11.8 and d) 21.1 wt. %.

Rate capability of the hybrid battery–capacitor

The discharge curves of the hybrid battery–capacitors LAC/ $\text{Li}_4\text{Ti}_5\text{O}_{12}$ and DMLAC/ $\text{Li}_4\text{Ti}_5\text{O}_{12}$ at different discharge current densities (the charge–discharge rates were the same for each case) are shown in Fig. 4. The specific capacities decreased slightly after 20 cycles when the hybrid battery–capacitors were discharged at several rates ranging from 2C to 10C. At the same rate, the capacity of the hybrid battery–capacitors increased when the content of LiFePO_4 in the com-

posite materials was increased. At 4C, with the *in-situ* synthesis, when the content of LiFePO₄ in LAC was 5.2, 11.8, 21.1, 28.5 and 34.2 wt. %, after 20 cycles, the capacity losses of the LAC/Li₄Ti₅O₁₂ hybrid battery-capacitor were 1.2, 1.0, 2.6, 4.4 and 4.8 %, respectively. Clearly, all the values were less than 4.8 %. However, when the direct mixing technique was used, after 20 cycles, the capacity loss were 2.0, 2.3, 1.9, 2.6 and 2.9 %, respectively, and noticeably, all the value were less than 2.9 %. These results demonstrate the hybrid battery-capacitors LAC/Li₄Ti₅O₁₂ and DMLAC/Li₄Ti₅O₁₂ with different LiFePO₄ content exhibited good performances at high charge-discharge rates, indicating that the preparation method of the composite cathode materials have almost no impact on the high rate charge-discharge performance of the hybrid battery-capacitors.

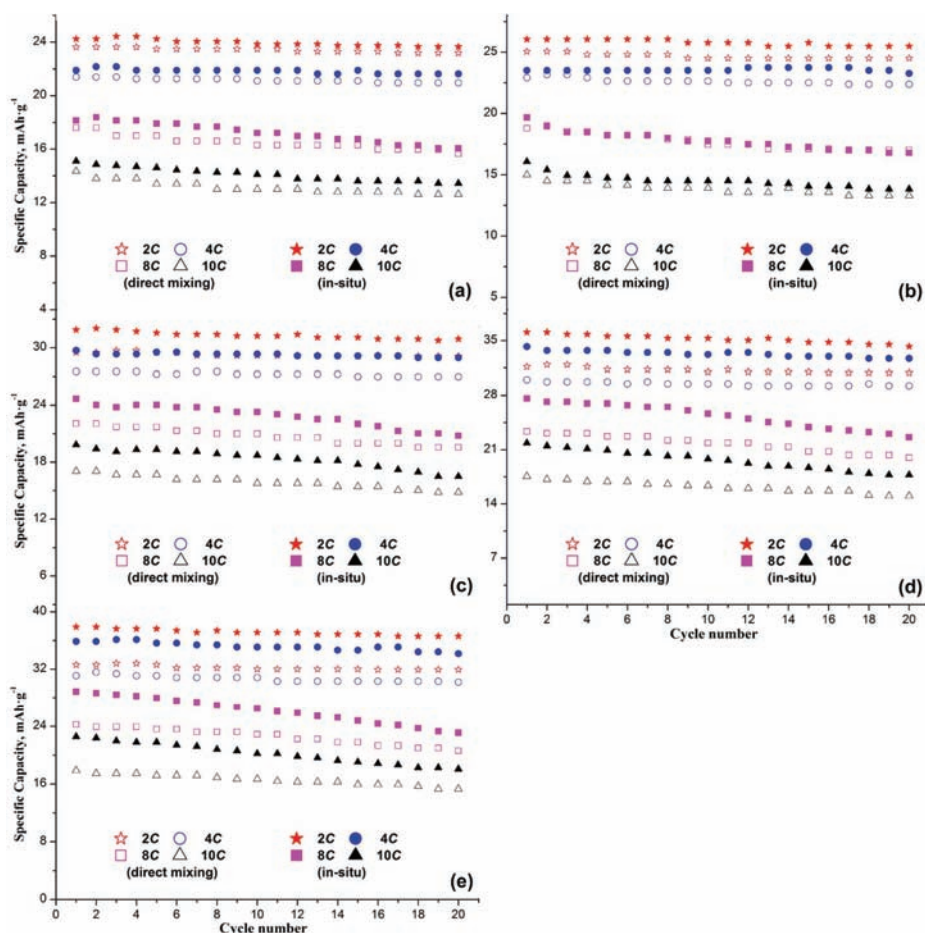


Fig. 4. Specific capacity of the hybrid battery-capacitors with different LiFePO₄ contents at different rates, where the specific capacity was calculated by the total mass of the active material in the hybrid battery-capacitors: a) 5.1, b) 11.8, c) 21.1, d) 28.5 and e) 34.2 wt. %.

Cycle performance of the hybrid battery–capacitor

A comparison of the discharge capacity between LAC/Li₄Ti₅O₁₂ and DMLAC/Li₄Ti₅O₁₂ hybrid battery–capacitors, an AC/Li₄Ti₅O₁₂ capacitor and a LiFePO₄/Li₄Ti₅O₁₂ battery during long-term cycling is shown in Fig. 5, for the same mass of cathode active material in each cell. In the *in-situ* synthesis, when the content of LiFePO₄ in the LAC was 5.2, 11.8, 21.1, 28.5 and 34.2 wt. %, the capacity loss of the LAC/Li₄Ti₅O₁₂ hybrid battery–capacitors was 8.6, 9.2, 10.1, 12.0 and 15.8 %, respectively, after 100 cycles. However, in direct mixing technique, the capacity loss was 8.5, 8.7, 9.6, 11.4 and 12.4 %, respectively, after 100 cycles. This indicates that the cycle performances of LAC/Li₄Ti₅O₁₂ and DMLAC/Li₄Ti₅O₁₂ were better than those of AC/Li₄Ti₅O₁₂ and LiFePO₄/Li₄Ti₅O₁₂ when the same cathode or anode was used in these cells. However, with increasing LiFePO₄ content, the cycle performances of the hybrid battery–capacitors LAC/Li₄Ti₅O₁₂ and DMLAC/Li₄Ti₅O₁₂ slightly decreased. At the same LiFePO₄ content, the capacity loss of DMLAC/Li₄Ti₅O₁₂ was less than that of LAC/Li₄Ti₅O₁₂, which was due to the better cycle performance of the commercial LiFePO₄-coated carbon or doped-carbon. These facts illustrate that the hybrid battery–capacitors LAC/Li₄Ti₅O₁₂ and DMLAC/Li₄Ti₅O₁₂ had good cycle performances. This also means that the preparation methods of the composite cathode materials had almost no impact on the high rate cycle performances of the hybrid battery–capacitors.

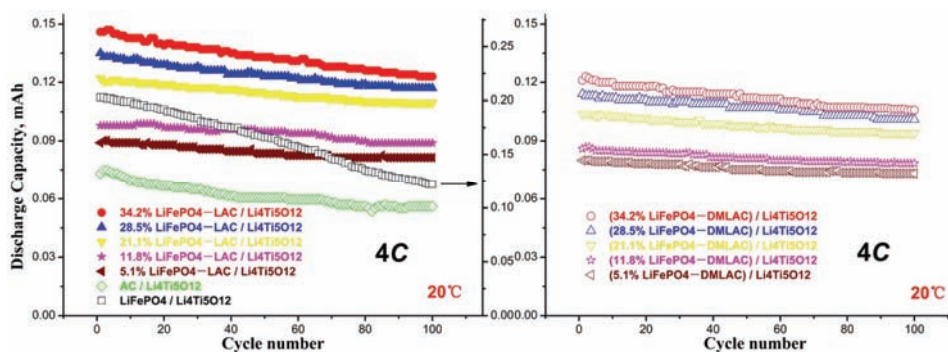


Fig. 5. Comparison of cycle performance of the LAC/Li₄Ti₅O₁₂ and DMLAC/Li₄Ti₅O₁₂ hybrid battery–capacitors, the AC/Li₄Ti₅O₁₂ capacitor and the LiFePO₄/Li₄Ti₅O₁₂ battery at a rate of 4C.

Comparison of the cathode materials performance

The results of the performances of the cathode materials (LAC, DMLAC composite electrodes and AC electrode) in different cell systems are given in Table III. Using the same electrolyte, when the content of LiFePO₄ in cathode composite was 5.2, 11.8, 21.1, 28.5 and 34.2 wt. %, the specific capacity of an LAC

single electrode in a LAC/Li₄Ti₅O₁₂ hybrid battery–capacitor was 11.6, 23.2, 44.6, 60.6 and 70.4 % higher than of that of an AC single electrode in an AC/Li₄Ti₅O₁₂ hybrid capacitor and an AC/AC electric double-layer capacitor, which was also about 87.8, 76.3, 69.2, 65.0 and 61.7 % of the maximum theoretical capacity. However, with a DMLAC single electrode, the specific capacity was 3.6, 8.4, 23.4, 35.4 and 41.6 % higher than that of that of an AC single electrode, which is also about 81.5, 67.1, 59.0, 54.8 and 51.3 % of the maximum theoretical capacity. These results indicate that the LAC and DMLAC composite cathode materials with different LiFePO₄ contents had high capacity characteristic. However, with increasing LiFePO₄ content, the capacity utilization of the hybrid battery–capacitors LAC/Li₄Ti₅O₁₂ and DMLAC/Li₄Ti₅O₁₂ slightly decreased. The above results illustrate that the preparation method of the composite cathode materials had almost no impact on the capacity performance of the composite cathodes.

TABLE III. Cathode performances of the different capacitors

Content of LiFePO ₄ wt. %	LAC		DMLAC		AC (AC/Li ₄ Ti ₅ O ₁₂)	AC (AC/AC)	
	(calculated)	(LAC/Li ₄ Ti ₅ O ₁₂)	(DMLAC/Li ₄ Ti ₅ O ₁₂)				
	C _g mA h g ⁻¹	Utilization rate, %	C _g mA h g ⁻¹	Utilization rate, %			
34.2	76.56	47.25	61.7	39.26	51.3	27.72	28.00
28.5	68.47	44.51	65.0	37.52	54.8		
21.1	57.96	40.08	69.2	34.22	59.0		
11.8	44.76	34.14	76.3	30.04	67.1		
5.1	35.24	30.95	87.8	28.71	81.5		

Cycle life performance of the hybrid battery–capacitor device

The results for the cycle life of the DMLAC/Li₄Ti₅O₁₂ hybrid battery–capacitor devices at a high rate are shown in Fig. 6. The hybrid battery–capacitor devices at a high rate provide high coulombic efficiencies close to 100 %. At 4C, when the contents of LiFePO₄ in the composite cathode were 15 and 22.5 wt. %, the capacity losses of the DMLAC/Li₄Ti₅O₁₂ hybrid battery–capacitor devices were 3.4 and 4.8 %, respectively, after 1000 cycles. Clearly, neither of the values was more than 4.8 %. However, at 8C, after 2000 cycles, the capacity losses were 8.1 and 9.6 %, respectively, but noticeably, neither of the values was more than 9.6 %. The results show that the DMLAC/Li₄Ti₅O₁₂ hybrid battery–capacitor devices had a good cycle life performance at a high rate.

All the above electrochemical testing data demonstrate that the preparation method of the composite cathode materials had almost no impact on rate capability, cycle performance or capacity performance of the composite cathodes. At 4C, when the content of LiFePO₄ in the cathode composite was 11.8, 21.1 and 28.5 wt. %, after 100 cycles the capacity losses of the LAC/Li₄Ti₅O₁₂ and DMLAC/

$\text{Li}_4\text{Ti}_5\text{O}_{12}$ hybrid battery–capacitors were less than 12.0 and 11.4 %, respectively. Moreover, the capacity and capacity utilization of the composite cathodes achieved better results. These facts illustrate that the overall electrochemical performance of the composite cathodes and the hybrid battery–capacitors was the best when the content of LiFePO_4 in the composite cathode materials ranged from 11.8 to 28.5 wt. %, while the preparation method of the composite cathode materials had almost no impact on the electrochemical performances of the composite cathodes and the hybrid battery–capacitors.

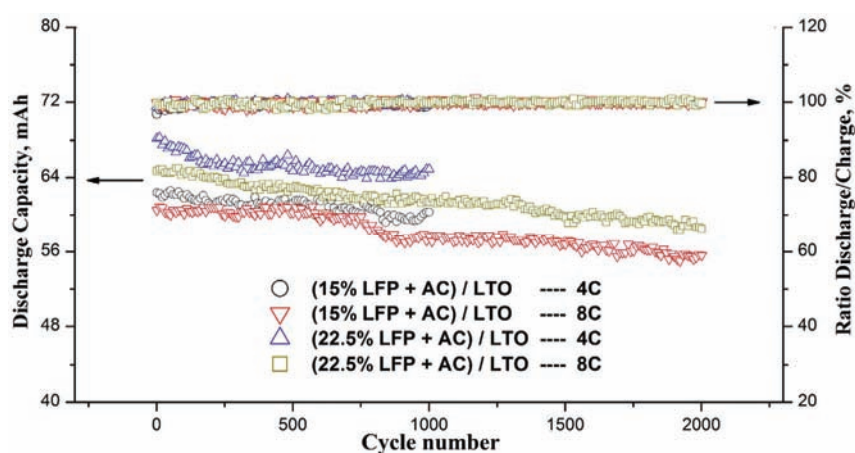


Fig. 6. Discharge capacity and discharge–charge ratio vs. number of cycle for the hybrid battery–capacitor devices at a high rate.

CONCLUSIONS

In this work, LAC and DMLAC composite cathode materials with different LiFePO_4 contents were synthesized by the *in-situ* synthesis method and the direct mixing technique, respectively, and then the hybrid battery–capacitors LAC/ $\text{Li}_4\text{Ti}_5\text{O}_{12}$ and DMLAC/ $\text{Li}_4\text{Ti}_5\text{O}_{12}$ with a $\text{Li}_4\text{Ti}_5\text{O}_{12}$ anode were assembled. The results showed that the hybrid battery–capacitors LAC/ $\text{Li}_4\text{Ti}_5\text{O}_{12}$ and DMLAC/ $\text{Li}_4\text{Ti}_5\text{O}_{12}$ had advantages of both a high rate capability and a high capacity, while the method of preparation of the composite cathode materials had almost no impact on the electrochemical performance of the hybrid battery–capacitors. Moreover, taking into account the capacity and capacity utilization, the overall electrochemical performance of the composite cathodes and the hybrid battery–capacitors was the best when the content of LiFePO_4 in the composite cathode materials ranges from 11.8 to 28.5 wt. %. More importantly, the hybrid battery–capacitor devices had a good cycle life performance at a high rate. When the content of LiFePO_4 in composite cathode was 15 and 22.5 wt. %, after 1000 cycles, the capacity losses of the DMLAC/ $\text{Li}_4\text{Ti}_5\text{O}_{12}$ hybrid battery–capacitor device at

4C were less than 4.8 %. However, at 8C, after 2000 cycles, the capacity losses were less than 9.6 %.

ИЗВОД

УТИЦАЈ САДРЖАЈА LiFePO₄ И НАЧИНА ДОБИЈАЊА НА СВОЈСТВА
(LiFePO₄ + AC)/Li₄Ti₅O₁₂ ХИБРИДА БАТЕРИЈА-КОНДЕНЗАТОРXUE BU HU¹, ZI JI LIN², LI LIU², YONG LIAN HUA¹ и ZHENG HUA DENG²

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In-situ техником и техником директног мешања синтетисана су два катодна композитна материјала, означена као LAC, односно DMLAC, који садрже LiFePO₄ и активирани угљеник (AC). Од њих су формиран LAC/Li₄Ti₅O₁₂ и DMLAC/Li₄Ti₅O₁₂ хибриди батерија-кондензатор. Испитиван је утицај садржаја LiFePO₄ и технике припремања на цикличне волтамограме, брзину процеса и број циклуса пуњење/пражњење хибридних извора. Резултати су показали да су најбоље електрохемијске карактеристике хибридног извора постигнуте са садржајем LiFePO₄ у катодном композитном материјалу у опсегу 11,8–28,5 мас. %, док начин припреме није имао утицаја. Хибридни уређај батерија-кондензатор имао је задовољавајући број циклуса пуњење/пражњење при великим брзинама овог процеса. Након 1000 циклуса, губитак капацитивности уређаја DMLAC/Li₄Ti₅O₁₂ при 4C брзини није био већи од 4,8 %, а након 2000 циклуса при 8C брзини не већи од 9,6 %.

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