Metal Incorporated Polymeric Nanodots Based Electrode Material for Fluorescent Supercapacitors

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In this article, a new class of fluorescent supercapacitor electrode material has been developed by electro-polymerization of Rhodamine b dye in presence of Potassium Ferrocyanide on pencil graphite electrode. The morphology and surface properties of plain polymeric Rhodamine nanodots and Potassium Ferrocyanide incorporated polymeric Rhodamine nanodots were analyzed. Optical and electronic properties of Potassium Ferrocyanide incorporated polymeric Rhodamine b nanodots was found to be superior in comparison with polymeric Rhodamine b nanodots. Potassium Ferrocyanide incorporated polymeric Rhodamine b nanodots shows an enhanced specific conductance (320.0 F/g), and better rate capability 3 to 4 times higher than the plain polymeric Rhodamine b nanodots modified graphite electrodes. The electrochemical activity of the fluorescent Potassium Ferrocyanide doped polymeric Rhodamine b nanodots was found to be easily tailored by changing the concentration of Potassium Ferrocyanide. The 2:1 molar ratio (Potassium Ferrocyanide and Rhodamine b) based polymeric nanodots shows excellent specific capacitance (434.8 F g⁻¹) and fluorescent maxima 580 nm in comparison to other mixing ratios. Potassium Ferrocyanide doped polymeric Rhodamine b nanodots possess excellent rate performance, high energy density of 150.06 Wh kg⁻¹ at a power density of 900 W kg⁻¹, and robust cycling durability, with 99.5% retaining in its specific capacitance even after 6500 cycles. Even after 8 months (40,000 cycles) of testing, the performance of the supercapacitor did not degrade. The excellent performance of the investigated fluorescent nanodots as electrode material could open up the application of supercapacitors to Internet of Things (IoT) applications.

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For decades, batteries have been the preferred storage device for portable electronics because of their ability to store energy. But batteries take a long time to discharge and recharge, which limits their ability to deliver power. Overcoming this power deficit is difficult and even newer battery technologies such as lithium ion are still a poor solution for high power applications. In applications demanding high power, over-engineering the battery will rarely be the right solution, and with typically result in increased size, weight, and cost, and reduced cycle life and energy. This power deficit is being stretched further by the explosion of Internet of Things (IoT) applications. These applications are usually wireless-enabled, and yet they demand ever smaller and more portable devices, with more features and functions. Wireless transmissions, even over very short distances, present a tremendous power challenge to the necessarily small batteries being used in IoT devices. Supercapacitors will be a critical enabling technology for the IoT, offering a unique combination of high power and high energy, in a thin, flat and very small package. Supercapacitors combine the energy storage properties of batteries with the power discharge characteristics of capacitors.

Quantum dots are considered to be promising for fluorescent supercapacitors because of its excellent electrical and optical properties in addition to the large surface area. In addition, quantum dots have proven to be a smart nanomaterial for the development of miniaturized energy storage systems with high power densities, fast charge-discharge rates, and longer lifetimes. Among the various reported electrochemical energy storage systems such as batteries and supercapacitors, supercapacitors were found to be much more efficient than batteries in terms of power delivery and inferior in energy density. The low energy density is attributed to poor accesses of electrolyte to the electrode surface. In this regard, development of high surface area porous nanomaterials such as carbon nanomaterials, mixed-metal oxides, conducting polymers and recently metal-organic framework, greatly improved the conductivity and increased the SCs performances. Electrodes coated by conducting polymers such as poly-pyrrole, polypyrrole, polyaniline and nanostuctured conducting polymers used have been used in energy storage system. These conducting polymers based supercapacitors energy storage mechanism work on fast reversible redox reactions. Thus nanostructured conducting polymer or conducting polymer nanomaterial composites based supercapacitors can be categorized as pseudo-capacitors (charge storage mechanism involving fast surface redox reactions). The pseudo-capacitors energy storage devices have been attractive because of their higher power density. A major drawback of pseudo-capacitors is the cycle stability. In order to achieve the enhanced performance in terms of energy density, specific capacitance, and cycle stability of supercapacitors, various hybrid nanocomposites like metal oxides/carbon nanotubes, graphene–conducting polymers, carbon nanotubes conducting polymers, and metal oxides–conducting polymers have been developed.

The nano-modified electrodes suffer from the sluggish rate of ion transport during the oxidation-reduction phase due to the high interfacial resistances and the inherent resistance of the binder materials. The performance of nanocomposites not only depends on active materials used for development of electrode material but also its morphology and interfacial properties. Direct growth method has been adopted to construct active nanomaterials directly on the electrodes for supercapacitors. Adopting electro-polymerization technique for the synthesis of active nanomaterial directly on electrode surface has been found to be a promising approach to overcome the above discussed limitations and improve the supercapacitors performance. Only a few efforts have been reported in the development of fluorescent supercapacitors. Liao et al., have developed fluorescent dye incorporated multi-walled carbon nanotubes based fluorescent fiber electrode. Herein, we report a new type of dye based quantum dots i.e. polymeric quantum dots (P-nanodots) used for development of fluorescent supercapacitor. The P-nanodots possess excellent semiconducting properties, amplified energy transfer, π-conjugated
Scheme 1. Schematic illustration of development of Fe-P-nanodots modified PGE based fluorescent supercapacitor.

structure, small size (>30 nm), good solvent dispersion, abundant surface group which not only enhances pseudo-capacitive properties but also increases the binding to the surface of electrodes through chemical bonds. These nanodots are more competent in comparison with organic dyes, and traditional quantum dots.43–47 Potassium Ferrocyanide when present along with conducting polymeric nanosphere shows better electrical conductivity, high specific surface area and pseudo-capacitance.48,49 Herein, Potassium Ferrocyanide used as iron source, is wrapped by polymer Rhodamine to form an interlocked stable structure i.e. polymeric Rhodamine b nanodots during electro-polymerization. On the basis of the recently reported work,50 potassium Ferrocyanide incorporated P-nanodots would have a positive effect on the morphology, surface area and pore size distribution of fluorescent materials which, in turn, will benefit the electrochemical performance.

Till date, the synthesis of Potassium Ferrocyanide incorporated dye based polymer dots (Fe-P-nanodots) by electro-polymerization technique and its applications for electrochemical fluorescent supercapacitors have never been reported. Supercapacitors have become ubiquitous at this point. Supercapacitors have a much higher power density than batteries, which means that they can both deliver and absorb energy from the load much faster than batteries. They provide everything from back-up power for mobile phones to extending the battery life of devices that sometimes need quick bursts of power like a digital camera’s zoom feature. These supercapacitors referred to as electrochemical double layer capacitors (EDLCs) come in a growing variety of shapes, size and applications. For supercapacitors to reach a wider set of applications, research has been focused on improving the electrode material so the supercapacitors can hold more charge. Such a motivation forms the basis of this study.

Experimental

Materials and electrochemical characterization.—All the chemical reagents used were analytical grade without further purification. The microscopic features of the as-prepared materials were observed with scanning electron microscopy (SEM). FT-IR spectra were investigated by FT-IR microscope. The porosity, specific surface area, and their porous-structure were analyzed by N₂ adsorption/desorption analysis under 77 K Autosorb 1C (Quantachrome, USA) instrument. Fluorescence spectra was recorded by Olympus Fluorescence microscope. The FTIR spectra were recorded on a Tensor 27, Bruker (Germany) machine. Cyclic voltammetry (CV) measurements were conducted by using a CHI660E electrochemical workstation in a potential range from −1.6 V to 2 V (vs. Ag/AgCl) at a scan rate of 0.1 mV s⁻¹ using a three electrode cell assembly, consisted of a platinum wire, Ag/AgCl (3.0 M KCl), and Fe-P-nanodots modified pencil graphite electrode (PGE) as counter, reference and working electrodes, respectively. Chrono-potentiometry charge-discharge tests were performed on the electrochemical workstation at the same potential range, while the current densities varied from 1 to 30 A g⁻¹. Electrochemical impedance spectroscopy (EIS) measurements were conducted on the electrochemical workstation and frequency limits were set at 1000 kHz to 0.01 Hz with 5 mV of voltage amplitude at the open-circuit potential. All the experiments were performed at room temperature (25 ± 1°C).

Electrochemical calculations.—In an ideal supercapacitor, developed by three electrode system, the specific capacitance (Cₛ), energy density (E) and power density (P) can be calculated by using following equations:

\[
C_s = \frac{I_d \times t_d}{V \times m}
\]  \[1\]

\[
E = \frac{0.5 \times C_s \times V^2}{3.6}
\]  \[2\]

\[
P = \frac{E \times 3600}{t_d}
\]  \[3\]

Where I_d is the discharge current, m is the mass of the material, t_d is the discharge time, and V is the potential window.
Table I. Physical properties of different types of electrochemically grown Fe-P-nanodots.

| Rhb: PFc molar ratios | Fluorescence emission maxima (nm) | Quantum Yield (%) | S BET (m$^2$ g$^{-1}$) | V (cm$^3$ g$^{-1}$) | Specific Capacitance (F g$^{-1}$) |
|----------------------|----------------------------------|-------------------|-------------------------|-----------------|-----------------------------|
| 1:0 (P-nanodots)     | 495                              | 10.4              | 95.0                    | 0.054           | 265.0                       |
| 1:1 (Fe-P-nanodots$_1$) | 545                             | 12.6              | 235.4                   | 0.0867          | 320.0                       |
| 2:1 (Fe-P-nanodots$_2$) | 580                             | 21.5              | 265.5                   | 0.1245          | 434.8                       |
| 3:1 (Fe-P-nanodots$_3$) | 535                             | 8.8               | 281.7                   | 0.2453          | 367.7                       |
| 4:1 (Fe-P-nanodots$_4$) | 505                             | 7.2               | 293.8                   | 0.3564          | 348.6                       |

Synthesis of Fe-P-nanodots.—Pre-treatment or electro-activation of graphite electrode is necessary before electro- polymerization step to remove any impurities. To do this, +1.0 V (vs Ag/AgCl) was applied for 120 s in 0.1 M phosphate buffer (pH 7.0). The Fe-P-nanodots were prepared by electrochemical polymerization of Rhodamine b in presence of Potassium Ferrocyanide using three-electrode system. In three electrode cell system, Rhodamine b (0.1 mM) solution was taken in presence of 0.1 mM Potassium Ferrocyanide (PFc) containing buffer solution (pH 7.0) as electrolyte. The Potassium Ferrocyanide incorporated polymeric Rhodamine b nanodots were electrochemically grown on the surface of graphite by cyclic voltammetry, 100 scan cycles at 75 mV/s scan rate under constant stirring at room temperature. After 30 minutes, electrode surface was rinsed with distilled water and kept dry at room temperature Fe-P-nanodots were collected by scraping the surface of graphite. Obtained nanodots were dissolved and kept dry at room temperature Fe-P-nanodots were collected by centrifuging at 1200 rpm at 20 minutes. Scheme 1 is a schematic illustration of development of Fe-P-nanodots modified pencil graphite electrode based supercapacitor.

Figure 1. (A) Fluorescence emission spectrum of the P-nanodots and Fe-P-nanodots and (B) Nitrogen adsorption-desorption isotherms (black color- P-nanodots; pink color- Fe-P-nanodots$_1$; red color-Fe-P-nanodots$_2$; yellow color-Fe-P-nanodots$_3$; green color-Fe-P-nanodots$_4$).
Fig. 2B reveals Fe-P-nanodots modified graphite surface. The spherical dots with size range (∼70 nm) grown on the surface can be seen. The incorporation of PFc can be seen in Fig. 2B, inset. The shiny particle embedded in the sphere of P-nanodots corresponds to iron metal (Fig. 2B inset). Surface morphology analysis of Fe-P-nanodots by TEM further confirms the formation of Fe-P-nanodots and the size of Fe-P-nanodots is observed to be less than 70 nm. As shown in TEM image Fig. 2C, the Fe-P-nanodots had rough spherical shapes and were well dispersed with diameters in the range of 30–50 nm.

Elemental analysis was done through EDAX to confirm the coating of Fe-P-nanodots on the bare PGE surface. Figs. 3A, 3B shows EDAX analysis of P-nanodots and Fe-P-nanodots. Spectra recorded for P-nanodots electrode (Fig. 3A) shows the presence of the carbon, oxygen and nitrogen element. In EDAX spectrum of Fe-P-nanodots, the presence of extra iron element peak along with carbon, nitrogen and oxygen confirms the formation of Fe-P-nanodots on the surface of graphite electrode (Fig. 3B). The weight percentage of nitrogen (25.53%) and carbon (57.23%) found to be increase in comparison to P-nanodots modified PGE.

Comparative EDAX analysis of P-nanodots modified graphite and Fe-P-nanodots modified graphite, supports the incorporation of PFc in the polymeric network of dye based P-nanodots. In the FTIR spectrum of P-nanodots modified PGE (Fig. 3C), the existence of −OH (3400 cm⁻¹), and C=O (1725 cm⁻¹) indicates the functional groups present on the surface of graphite electrode after treatment with nitric acid. The existence of band at 1590 cm⁻¹ (-C-N), 1470 cm⁻¹ (-CH₂-) and 1340 cm⁻¹ (-C≡C-) indicates the electro-deposition of Rhb on graphite electrode. As depicted in the spectra (Fig. 3D), the Fe-P-nanodots possesses the characteristic bands of cyanide -CN (2120 cm⁻¹) corresponds to Potassium Ferrocyanide along with PRhb confirms the co-ordination of Potassium Ferrocyanide in the nanostructure of P-nanodots.

Electrochemical performance.—The capacitive behavior of electro-polymerized Fe-P-nanodots and P-nanodots modified PGE were determined by studying the electrochemical properties. The electrochemical properties of the Fe-P-nanodots modified PGE and P-nanodots electrode were evaluated by cyclic voltammetry (CV), chrono-potentiometry, charge-discharge cycles, and electrochemical impedance spectroscopy (EIS) in 0.1 M KOH aqueous electrolyte at pH 3.5. The CV plots of bare PGE, P-nanodots modified PGE and Fe-P-nanodots modified PGE are shown in Fig. S3. It is interesting to note that CV curve of Fe-P-nanodots modified PGE was found to be rectangular indicative of high specific surface area in the potential region of −1.6 to 2.0 V compared to P-nanodots modified PGE and bare PGE. The area of rectangular plot is a measure of the capacitance which is found to be higher in Fe-P-nanodots in comparison to P-nanodots modified PGE and bare PGE. Moreover, the Fe-P-nanodots modified PGE exhibits high specific capacitance (434.0 F g⁻¹) at a scan rate of 2 mV s⁻¹) in comparison to controlled P-nanodots (265 F g⁻¹, at a scan rate of 2 mV s⁻¹) and other reported metal hexacyanoferrate composites electrodes.54

The CV measurements were done at different potential window and scan rates, to characterize the behavior of PFc incorporated
P-nanodots. As revealed from Figs. 4A–4B, CV curves of Fe-P-nanodots was approximately quasi-reversible rectangle in a wide potential window in $-1.6 \, \text{V}$ to $2.0 \, \text{V}$. The redox peaks in CV and wide potential window corresponds to variable vacancy of PFc present in the network of the P-nanodots. The CV curves of Fe-P-nanodots indicates the pseudo-capacitance behavior of Fe-P-nanodots, seems to be associated with intercalation/de-intercalation of $K^+$ ions from electrolyte, along with reversible redox reactions. The tentative redox mechanism is speculated as follows: The $Fe^{2+}$ ions from $K_4[Fe(CN)_6]$ will be free in acidic medium pH 3.5 and oxidizes into $Fe^{3+}$. The released electrons along with hydrogen ions partially reduce the oxygen containing groups of graphite. The released $Fe^{3+}$ interacts with Rhb and adsorbed on the Rhb through electrostatic interactions. Furthermore, these $Fe^{3+}$ also interacts with $[Fe(CN)_6]$ which helps in formation of nanodots structure. The tentative reactions could be described as follows:

$$\text{Graphite} + \text{Rhb} + \text{PFc} \rightarrow \text{Graphite} - \text{Fe} - \text{P} - \text{nanodots}$$

$$\text{Graphite} + n\text{H}^+ + n\text{e}^- \rightarrow \text{Graphite} + n\text{H}_2$$

$$K^+ + Fe^{3+} + [Fe(CN)_6]^{4-} \leftrightarrow K[Fe(CN)_6]$$

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In an electrochemical process, a b-value of 0.5 confirms a diffusion controlled process, whereas 1.0 indicates a capacitive process or non-diffusion-controlled process. It is concluded that for pseudo-capacitive electrode materials b value should be $\sim 1.0$. Thus, intercalation pseudo-capacitive electrode materials can be characterized by a linear dependence on scan rate or measured current.

In present case of Fe-P-nanodots modified PGE, the slope of the plot of log ($i$) versus log ($v$) was found to be $\sim 0.97$ for both potential ranges ($\sim b = 1$), indicating the Fe-P-nanodots modified PGE exhibits the intercalation pseudo-capacitance behavior and fast electrolyte ions $K^+$ insertion process (Figs. 4E–4F). In addition, the charge storage process in Fe-P-nanodots was found to be surface-controlled and non-diffusive limited, which provides superior rate capability. The $K^+$ intercalation pseudo-capacitive behavior of the Fe-P-nanodots can be attributed to the unique interconnected nanodots structure that makes more accessible channels for $K^+$ insertion/extraction in the Fe-P-nanodots. The specific capacitance of the Fe-P-nanodots at different potential window (current density 1 A g$^{-1}$) was calculated from the CVs as a function of scan rate (shown in Figs. 4G–4H). In both cases, the specific capacitance was found to decrease with increase in scan rate because of the progress of intercalation of electrolyte ions into the network of Fe-P-nanodots. The results obtained in both cases confirms

$$i = av^b$$

Here, $b$ value can be find by slope of the plots between log ($i$) and log ($v$). In an electrochemical process, a b-value of 0.5 confirms a diffusion controlled process, whereas 1.0 indicates a capacitive process or non-diffusion-controlled process. It is concluded that for pseudo-capacitive electrode materials b value should be $\sim 1.0$. Thus, intercalation pseudo-capacitive electrode materials can be characterized by a linear dependence on scan rate or measured current.

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Figure 4. CV curves of Fe-P-nanodots modified PGE obtained from (A) \(-1.6 \text{ to } 2.0 \text{ V}\) and (B) \(-1.5 \text{ to } 2.0 \text{ V}\) at a scan rate of \(15 \text{ mV s}^{-1}\), and (C, D) different scan rates from 5 to \(300 \text{ mV s}^{-1}\) in 0.1 M KOH electrolyte. The logarithm of current versus logarithm of scan rates (E, F) and specific capacitance as a function of the potential scan rates (G, H) for different potential windows.
that the charge storage in proposed fluorescent supercapacitors follows intercalation pseudo-capacitive mechanism.57

The high rate capability or cyclic stability is an important parameter for energy storage systems. For P-nanodots specific capacitance remains 100% for 2500 cycles whereas in Fe-P-nanodots, specific capacitance remains constant for 6500 charging-discharging cycles at 5 A g\(^{-1}\). This comparative studies results demonstrate that the Fe-P-nanodots shows an almost 2.5 times higher cycling stability than P-nanodots. This comparative result again revealed that incorporation of PFc in P-nanodots enhances the capacitive performance. PFc became a part of nanostructured P-dots which changed the quantum property as well as electronic property of P-nanodots. In this work cyclic stability was measured at two different current density 5 A g\(^{-1}\) and 10 A g\(^{-1}\) Fig. 5A. Specific capacitance was found to be decrease with increase in current density value.

In both cases, the specific capacitance value retained for 100% up to 6500 cycles, revealing firm bonding between PFc and Rhb in polymer network. The specific capacitance is remarkably higher than previously reported supercapacitors based on Potassium Ferrocyanide, conducting polymer based electrodes and quantum dots as highlighted in Table II. To further explore the electronic performance of Fe-P-nanodots, other two important electrochemical parameters such as power density and energy density were evaluated. Fig. 5B shows the Ragone plot of the Fe-P-nanodots modified PGE obtained in the voltage window of –1.6 V to 2.0 V. It is noted that our device delivered an energy density of 150.06 Wh kg\(^{-1}\) at a power density of 90 W kg\(^{-1}\). In addition, at a power density as high as 9565 W kg\(^{-1}\), the energy density still can be retained at 80 Wh kg\(^{-1}\). The significant enhancement in energy densities of fluorescent supercapacitor can be attributed to the wide potential window and high capacitive behavior of the Fe-P-nanodots based fluorescent supercapacitor. In addition, the proposed supercapacitor exhibits 2 to 3 times in terms of specific capacitance, energy density as well as power density, than that of recently reported Polymer dots/graphene, Graphene Quantum Dots on Halloysite Nanotubes and iron oxide-carbon nanotube based supercapacitor.46,61 Supercapacitors are going to power up Internet of Things (IoT). The devices that make up the IoT will likely depend on some kind of energy-harvesting mechanism that will make the incorporation of small but powerful energy storage devices like supercapacitors a critical element of these devices. The investigated electrode material can bring super capacitors closer to reality in powering up IoT devices.

Conclusions

We have successfully developed a new fluorescent supercapacitor through simple and easy electrochemical polymerization method. The Rhodamine b dye and Potassium Ferrocyanide optimal molar ratio (2:1) was used to prepare the polymer nanodots. The nanodots modified pencil graphite electrode showed excellent capacitive and fluorescent properties. The electrochemically prepared P-nanodots and Fe-P-nanodots exhibited 265 F g\(^{-1}\) and 434.8 F g\(^{-1}\) at 1 A g\(^{-1}\) and a capacitance retention over 99.5% after 40,000 charging and discharging cycles, respectively. Additionally, the Fe-P-nanodots supercapacitor possess a high energy density 150.06 Wh kg\(^{-1}\) at a power density of 900 W kg\(^{-1}\). Such a supercapacitive behavior of Fe-P-nanodots modified pencil graphite electrode is attributed to the incorporation of PFc in P-nanodots network which improves the faradaic reaction and intercalation/de-intercalation of electrolyte ions. The results from this study will enable the advancement of supercapacitors toward powering up IoT devices. A super capacitor can collect energy from weak energy sources over a long time, and supply that charge to a high-current load for a short time – a wireless transmit burst for example, or for data storage on power fail. These features make supercapacitors ideal for IoT applications.

Table II. The comparison of the specific capacitance Fe-P-nanodots and the previously reported supercapacitors in the literature.

| Electrode material | Specific Capacitance (F g\(^{-1}\)) | Reference |
|--------------------|-------------------------------------|-----------|
| Iron oxide incorporated carbon nanotube | 18.0 | 18 |
| Fluorescent polypyrrole nanospheres | 245.8 | 41 |
| Polymer dots/graphene sheets composites | 364.2 | 47 |
| Graphene quantum dots | 110.0 | 58 |
| MWNT incorporated polyaniline composite film | 233.0 | 59 |
| Polypyrrole coated carbon nanotube | 179.0 | 60 |
| Graphene Quantum Dots on Halloysite Nanotubes | 363.0 | 61 |
| Carbon quantum dots/polyprrole composite film | 308.0 | 62 |
| Carbon quantum dots/polyprrole composite film on C\(_60\) molecules | 157.4 | 63 |
| Fe-P-nanodots | 434.0 | Present work |

Figure 5. (A) Cycle stability of Fe-P-nanodots modified PGE at various current densities and (B) energy density as a function of power densities.

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