Notice

DESCRIPTION TWI344940B

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This invention relates to a method for manufacturing nano-sized iron oxide rods, and more particularly to a method for manufacturing nano-sized iron oxide rods that can be bonded to organic or inorganic molecules. With the rapid development of nanotechnology, various nanomaterials with nanostructures are being used more and more widely in various fields. In order to meet the requirements of miniaturization, high density and low power consumption of various components, the manufacturing process of various nanomaterials is constantly being improved.

本發明是有關於一種奈米級氧化鐵棒之製造方法，且特別是有關於一種可與有機或無機分子結合的奈米級氧化鐵棒之製造方法。 隨著奈米科技的日新月異，各種具有奈米結構的奈米材料在各領域的應用愈趨廣泛。 為了滿足各類元件於小型化、高密度、低功耗等需求，各類奈米材料的製程亦不斷地進行改良。

There are two common methods for preparing nano-sized iron oxide rods: the sol-gel method and the oxidation method.

習用製備奈米級氧化鐵棒的方法有溶膠－凝膠法(Sol－Gel Method)及氧化法(Oxidation Method)兩種。

The sol-gel method is used to prepare nano-sized iron oxide rods through hydrolysis and condensation. This method uses specific nanomaterials as precursors. After hydrolysis, the precursors form a sol, which is then condensed to remove moisture and form a gel. Finally, a low-temperature heat treatment is applied to generate multiple nanoscale iron oxide rods.

溶膠凝膠法係以水解、縮合的方式，進行奈米級氧化鐵棒的製備。 此法採用特定的奈米材料作為前驅物，此前驅物水解後形成溶膠(Sol)，再經由縮合去除水分而形成凝膠(Gel)，最後加以低溫熱處理即可生成複數個奈米級氧化鐵棒。

Although this sol-gel method can produce a large number of nano-sized iron oxide rods, it cannot be directly fixed and grown on the substrate. An adhesive must be mixed separately to fix the generated nano-sized iron oxide rods on the substrate.

此溶膠凝膠法雖可大量產出奈米級氧化鐵棒，但卻無法直接固定生長於基材上，需另外混合接著劑才可將生成之奈米級氧化鐵棒固定於基材上。

While the conventional oxidation method can directly generate nano-sized iron oxide rods on the substrate, this method requires higher temperatures, approximately 600 to 800 degrees Celsius, and the constant temperature time during heating needs to be maintained for tens of hours. Furthermore, oxygen needs to be introduced as the atmosphere for the heating reaction, so the safety of the process cannot be reduced. In addition, the cost of pure iron used as the base material is also very high.

而習用之氧化法雖可直接在基材上生成奈米級氧化鐵棒，但此法所需的溫度較高，約需攝氏600至800度，加熱時的恆溫時間則需維持長達數十小時，又需通入氧氣作為加熱反應用的氣氛，故其製程安全性無法降低。 另外，作為基材而使用的純鐵材料成本亦甚高。

Therefore, many research teams currently working on the preparation of nano-sized iron oxide rods are dedicated to developing methods for preparing nano-sized iron oxide rods that are simple to manufacture, low-temperature, low-cost, and highly safe.

因此，目前關於製備奈米級氧化鐵棒的諸多研究團隊均致力於開發製程簡單化、低溫、低成本與高安全性製備奈米級氧化鐵棒的方法。

To address the aforementioned and other problems and to achieve the technical advantages claimed by this invention, this invention provides a method for manufacturing nano-sized iron oxide rods. Therefore, the purpose of this invention is to provide a method for manufacturing nano-sized iron oxide rods with a simplified process, lower cost, and higher safety.

為解決上述和其他的問題，並且達到本發明所主張的技術優點，本發明提供一種奈米級氧化鐵棒之製造方法因此本發明的目的就是在提供一種奈米級氧化鐵棒之製造方法，以較簡化之製程、較低廉之成本及較高的安全性製備奈米級氧化鐵棒。

In accordance with the above-mentioned objectives of this invention, a simplified method for manufacturing nano-sized iron oxide rods is proposed. According to a preferred embodiment of this invention, polymer materials and compounds are mixed into a solution in a specific weight ratio.

根據本發明之上述目的，提出一種較簡化製程的奈米級氧化鐵棒之製造方法，依照本發明一較佳實施例，將高分子材料與化合物依照特定的重量比例混合成溶液。

Heat and stir the solution simultaneously until it becomes a viscous mixture.

加熱並同時攪拌此溶液，直至溶液變成黏稠狀之混合物。

This viscous mixture is evenly coated onto a substrate to form a precursor. The precursor is then heated in a high-temperature furnace to grow multiple nanoscale iron oxide rods.

將此黏稠狀混合物均勻塗佈於基材上，形成前驅物，再將此前驅物置於高溫爐中加熱，即可成長出複數個奈米級氧化鐵棒。

In accordance with the purpose of this invention, a method for manufacturing nano-sized iron oxide rods with lower cost and higher safety is proposed. According to a preferred embodiment of this invention, a substrate is placed in a device for cleaning, the device containing a solution for cleaning the substrate.

根據本發明之目的，提出一種較低成本與較高安全性的奈米級氧化鐵棒之製造方法，依照本發明一較佳實施例，將基材置於裝置中清洗，此裝置中含有清洗基材用的溶液。

The polymer material and the metal catalyst compound are mixed in a certain weight ratio, and then mixed with distilled water to form an aqueous solution.

把高分子材料與金屬觸媒化合物依照一重量比例混合，再與蒸餾水混合成水溶液。

Heat and stir the aqueous solution until it becomes a viscous mixture. Then, evenly spread the viscous mixture onto the substrate to form a precursor, and then perform a drying process at room temperature.

加熱並攪拌此水溶液，直到水溶液變成黏稠狀之混合物，再將黏稠狀混合物均勻塗佈於基材上形成前驅物後，在室溫下進行乾燥製程。

Finally, the precursor is placed in a high-temperature furnace and heated. Under specific reaction and heating conditions, multiple nanoscale iron oxide rods of a certain size can be grown in a controlled manner.

最後將前驅物置於高溫爐中加熱，經由特定的反應升溫條件，即可控制成長出一定尺寸之複數個奈米級氧化鐵棒。

Referring to Figure 1, which illustrates a flowchart of a method 100 for manufacturing a nano-sized iron oxide rod according to a preferred embodiment of the present invention.

參照第1圖，其繪示依照本發明一較佳實施例的一種奈米級氧化鐵棒製造方法100之流程圖。

This manufacturing method 100 includes the following steps: Step 110 involves mixing the polymer material polyethylene glycol (PEG) and the compound ferric sulfate (Fe2(SO4)3・nH2O) in a weight ratio to form a solution, wherein the weight ratio ranges from 1 to 1000.

此製造方法100包含以下步驟：步驟110係將高分子材料聚乙二醇(Polyethylene Glycol；PEG)與化合物硫酸鐵(Ferric Sulfate Hydrate；Fe2(SO4)3．nH2O)依照一重量比例混合成一溶液，此重量比例的比值範圍為1至1000。

Step 120 involves heating the solution with the first device and stirring until the solution becomes a viscous mixture.

步驟120係以第一裝置加熱此溶液，並攪拌至溶液變成黏稠狀之混合物。

Step 130 involves uniformly applying this viscous mixture onto the substrate to form a precursor.

步驟130係將此黏稠狀混合物均勻塗佈於基材上，形成前驅物。

Step 140 involves placing the previously prepared material in a second device and heating it to grow multiple nano-sized iron oxide rods.

步驟140係將此前驅物置於第二裝置中加熱，即可成長出複數個奈米級氧化鐵棒。

In step 110, polyethylene glycol (PEG) serves as the carbon source for the nano-sized iron oxide rods, and the compound can also be iron(III) oxide hydrate (FeO(OH)) or ferric nitrate hydrate (Fe(NO3)3・9H2O).

步驟110中的聚乙二醇(Polyethylene Glycol；PEG)係作為奈米級氧化鐵棒的碳來源，而化合物亦可為氫氧化鐵(Iron(III)Oxide Hydrate；FeO(OH))或硝酸鐵(Ferric Nitrate Hydrate；Fe(NO3)3.9H2O)。

The first device used to heat the solution in step 120 can be a hot plate, which is set to a temperature of about 70 degrees Celsius and simultaneously stirs the solution until it becomes viscous.

步驟120中加熱溶液用的第一裝置可為加熱攪拌器(Hot Plate)，此加熱攪拌器的溫度設定約為攝氏70度，並同時進行攪拌動作直至溶液成黏稠狀。

In step 130, the viscous solution is evenly applied to the substrate to form a precursor, and then heated after it dries.

步驟130中將黏稠狀溶液均勻塗佈至基材上形成之前驅物，待其乾燥後再進行加熱。

The second device for heating the precursor in step 140 can be a high-temperature furnace, through which multiple nano-sized iron oxide rods can be grown by setting reaction heating conditions.

步驟140中加熱前驅物用的第二裝置可為高溫爐，經由設定的反應昇溫條件，即可成長出複數個奈米級氧化鐵棒。

The reaction heating conditions of this high-temperature furnace are a heating rate of 20 degrees Celsius per minute (20°C/min), a reaction temperature of 350 to 750 degrees Celsius, and a constant temperature time of 4 hours.

此高溫爐的反應昇溫條件為昇溫速率為每分鐘上昇攝氏20度(20°C/min)，反應溫度為攝氏350至750度，恆溫時間則為4小時。

Referring to Figure 2, which illustrates a flowchart of a method 200 for manufacturing nano-sized iron oxide rods according to a preferred embodiment of the present invention.

參照第2圖，其繪示依照本發明一較佳實施例的一種奈米級氧化鐵棒製造方法200之流程圖。

This manufacturing method 200 includes the following steps: Step 210 involves placing the substrate in a third device for cleaning, the third device containing a solution for cleaning the substrate.

此製造方法200包含以下步驟：步驟210係將基材置於第三裝置中清洗，此第三裝置中含有清洗基材用的溶液。

Step 220 involves mixing the polymer material polyethylene glycol (PEG) and the metal catalyst compound ferric sulfate ($Fe_2(SO_4)_3 \cdot nH_2O$) in a weight ratio (the weight ratio ranges from 1 to 1000), and then mixing them with distilled water to form an aqueous solution.

步驟220係將高分子材料聚乙二醇(Polyethylene Glycol；PEG)與金屬觸媒化合物硫酸鐵(Ferric Sulfate Hydrate；$Fe_2(SO_4)_3 \cdot nH_2O$)依照一重量比例混合(此重量比例的比值範圍為1至1000)，再與蒸餾水混合成水溶液。

Step 230 involves heating the aqueous solution with the fourth device and stirring the aqueous solution to form a viscous mixture.

步驟230係以第四裝置加熱水溶液，並攪拌此水溶液成黏稠狀之混合物。

Step 240 involves uniformly coating the viscous mixture onto the substrate using the fifth device to form a precursor, followed by a drying process.

步驟240係以第五裝置將該黏稠狀混合物均勻塗佈於基材上形成前驅物後，進行乾燥製程。

Step 250 involves placing the precursor in the sixth device and heating it to grow multiple nano-sized iron oxide rods.

步驟250係把前驅物置於第六裝置中加熱，即可成長出複數個奈米級氧化鐵棒。

The substrate in step 210 can be carbon steel sheet or pure iron sheet with a purity of 99.99%. The carbon steel sheet is a low-carbon steel material, which is much cheaper than pure iron sheet.

步驟210中的基材可為碳鋼片或純度為99.99%的純鐵片，其中的碳鋼片為低碳鋼材料，成本遠較純鐵片為低。

The solution used for cleaning the substrate can be ethanol, and the third device can be an ultrasonic oscillator.

清洗基材用的溶液可為乙醇，且第三裝置可為超音波震盪器。

In step 220, polyethylene glycol serves as the carbon source for the nano-sized iron oxide rods, while the metal catalyst compound can also be iron(III) oxide hydrate (FeO(OH)) or ferric nitrate hydrate (Fe(NO3)3・9H2O).

步驟220中的聚乙二醇係作為奈米級氧化鐵棒的碳來源，而金屬觸媒化合物亦可為氫氧化鐵(Iron(III) Oxide Hydrate；FeO(OH))或硝酸鐵(Ferric Nitrate Hydrate；Fe(NO3)3．9H2O)。

將前驅物置於室溫下。

The fourth device for heating the aqueous solution in step 230 can be a heating stirrer. The temperature of this heating stirrer is set to approximately 70 degrees Celsius, and it stirs the solution until it becomes a viscous mixture.

步驟230中加熱水溶液之第四裝置可為加熱攪拌器，此加熱攪拌器的溫度設定約為攝氏70度，並同時進行攪拌動作直至水溶液變成黏稠狀之混合物。

The fifth device in step 240 can be a spin coater, which can uniformly coat a viscous mixture onto a substrate to form a precursor.

步驟240中的第五裝置可為旋轉塗佈機(Spin Coater)，此旋轉塗佈機可將黏稠狀混合物均勻塗佈於基材上形成前驅物。

Place the precursor at room temperature and allow it to dry until the weight of the precursor no longer decreases.

將前驅物置於室溫下，進行乾燥製程直至此前驅物重量不再減少為止。

The sixth device in step 250 can be a high-temperature furnace. When the high-temperature furnace operates under the set reaction heating conditions, the polymer compounds and metal catalyst compounds in the precursor will undergo solvent removal, hydrogen removal and thermal decomposition, thereby reacting to generate multiple nano-sized iron oxide rods.

步驟250中的第六裝置可為高溫爐，當高溫爐隨著設定的反應昇溫條件動作時，前驅物中的高分子化合物與金屬觸媒化合物會進行去溶劑、除氫及熱裂解等作用，進而反應生成複數個奈米級氧化鐵棒。

The reaction heating conditions of this high-temperature furnace are a heating rate of 20 degrees Celsius per minute (20°C/min), a reaction temperature of 350 to 750 degrees Celsius, and a constant temperature time of 4 hours.

此高溫爐的反應昇溫條件為昇溫速率為每分鐘上昇攝氏20度(20°C/min)，反應溫度為攝氏350至750度，恆溫時間則為4小時。

Referring to Figure 3, this image is a photograph taken with a scanning electronic microscope (SEM).

參照第3圖，此圖為掃瞄式電子顯微鏡(Scanning Electronic Microscope；SEM)所拍攝之照片。

As shown in the figure, the diameters of the various nano-sized iron oxide rods generated on the substrate range from approximately 8 to 70 nanometers.

由圖中可知，在基材上所生成之各種直徑的奈米級氧化鐵棒，其直徑分佈約從8到70奈米。

By changing the reaction heating conditions of the high-temperature furnace, the diameter of the generated nano-sized iron oxide rods can be controlled.

當改變高溫爐之反應升溫條件時，可藉以控制生成奈米級氧化鐵棒之直徑。

As can be seen from the above preferred embodiments of the present invention, the present invention has the following advantages compared with conventional methods:

由上述本發明較佳實施例可知，本發明與習用相較具有下列優點：

1.

The process operates at a low reaction temperature, requiring only 300 to 400 degrees Celsius, far lower than the commonly used 600 to 800 degrees Celsius, thus significantly reducing the energy consumption required.

製程的反應溫度低，僅需攝氏300至400度，遠較習用的攝氏600至800度為低，故所需耗費之能源亦降低許多。

2.

Polymer materials used as carbon sources for nano-sized iron oxide rods are inexpensive, and low-carbon steel materials used as substrates are also much cheaper than pure iron materials, so the overall material cost is more economical than conventional materials.

作為奈米級氧化鐵棒碳源之高分子材料價格低廉，且作為基材之低碳鋼材料亦比純鐵材料便宜甚多，故整體物料成本也較習用節省。

3.

Because polymer materials and metal catalyst compounds can be dissolved in distilled water at room temperature, the preparation process of the precursor is quite simplified.

因高分子材料與金屬觸媒化合物在常溫下即可溶於蒸餾水，使前驅物的製備過程相當簡化。

4.

When the precursor is heated in a high-temperature furnace, no special atmosphere is needed to control the reaction, and the reaction time is shorter. Compared with the conventional method of requiring oxygen and long-term high-temperature reactions, the safety of the process is greatly improved.

前驅物置於高溫爐中加熱反應時，不需通入特別的氣氛以控制反應，且反應時間較短，與習用需通入氧氣且常時間高溫反應相較之下，製程的安全性大為提高。

5.

This invention can produce nanoscale iron oxide rods with a diameter distribution of 8 to 70 nanometers, which is much smaller than the 30 to 70 nanometers produced by conventional oxidation methods. Furthermore, nanoscale iron oxide rods of different diameters can be grown by temperature control.

本發明可生成直徑分佈為8至70奈米之奈米級氧化鐵棒，與習用氧化法之30至70奈米相比還小許多，且經由溫度控制可生長不同直徑之奈米級氧化鐵棒。

Although the present invention has been disclosed above with reference to a preferred embodiment, it is not intended to limit the present invention. Anyone skilled in the art may make various modifications and alterations without departing from the spirit and scope of the present invention. Therefore, the scope of protection of the present invention shall be determined by the appended patent application.

雖然本發明已以一較佳實施例揭露如上，然其並非用以限定本發明，任何熟習此技藝者，在不脫離本發明之精神和範圍內，當可作各種之更動與潤飾，因此本發明之保護範圍當視後附之申請專利範圍所界定者為準。

100... Manufacturing Method

100・・・製造方法

110... Steps

110・・・步驟

120... Steps

120・・・步驟

130... Steps

130・・・步驟

140... Steps

140・・・步驟

200・・・ Manufacturing Method

200・・・製造方法

210・・・Steps

210・・・步驟

220・・・Steps

220・・・步驟

230・・・Steps

230・・・步驟

240... Steps

240・・・步驟

250... steps

250・・・步驟

To make the above and other objects, features, advantages and embodiments of the present invention more apparent, the accompanying drawings are described in detail below: Figure 1 is a flowchart (a) illustrating a method for manufacturing nano-sized iron oxide rods according to a preferred embodiment of the present invention.

為讓本發明之上述和其他目的、特徵、優點與實施例能更明顯易懂，所附圖式之詳細說明如下：第1圖係繪示依照本發明一較佳實施例的一種奈米級氧化鐵棒製造方法之流程圖(一)。

Figure 2 is a flowchart (II) illustrating a method for manufacturing nano-sized iron oxide rods according to a preferred embodiment of the present invention.

第2圖係繪示依照本發明一較佳實施例的一種奈米級氧化鐵棒製造方法之流程圖(二)。

Figure 3 is a photograph taken with a scanning electron microscope of the nanoscale iron oxide rod produced by this invention.

第3圖係為掃瞄式電子顯微鏡拍攝本發明生成的奈米級氧化鐵棒之照片。