Journal of Materials and
Environmental Sciences
ISSN : 2028-2508

JMES, 2017 Volume 8, Issue 5, Page 1654-1660

http://www.jmaterenvironsci.com/

Ceramic Matrix Composites: Processing Techniques and Recent advancements

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Received 20 Aug 2016,
Revised 18 Feb2017,
Accepted 20 Feb 2017

Keywords

- ✓ Sol-gel;
- ✓ Co-precipitation;
- ✓ Plasma spraying;
- ✓ Surfactant assisted;

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Abstract

Ceramic matrix composites (CMCs) have gained industrial importance due to their high thermal and mechanical properties as compared to single phase ceramic materials. Various properties such as thermal conductivity, mechanical strength and hardness etc. have been improved due to the utilization of ceramic material as both for matrix and reinforcement. Processing route influences the properties of ceramic matrix composites. The present investigation reports different processing routes for synthesis of various kinds of ceramic matrix composites. Surfactant assisted hydrothermal route has been utilized to achieve powder of alumina-titania composite in nano size range (25-27 nm). Phase formation and particle size of prepared powder has been investigated by X-ray diffraction (XRD) and Field emission scanning electron microscopy (FESEM).

1. Introduction

Composites are advanced engineering materials having combination of two or more constituents whether organic, inorganic or any metal which will give the combined properties of each phase [1]. Generally, a composite material consists of two phases. One is matrix (major phase) and another is reinforcement (minor phase and dispersed into matrix). The matrix phase holds and transfers the load to reinforced phase whereas reinforcement phase helps in providing strength and stiffness to composite. Composite materials are classified mainly into five different groups: 1. Metal matrix composite, 2. Ceramic matrix composite 3. Polymer matrix composite 4. Carbon carbon composite and 5. Hybrid matrix composite [2]. The use of these different composites is based on the application requirement. Among all, ceramic matrix composites (CMCs) have gained a significant importance due to the improved properties as compared to the conventional or traditional ceramics. They are developed to overcome the lack in reliability of single phase ceramic materials. Ceramic matrix composites include the reinforcement of ceramic material in a ceramic matrix to get superior properties. The dispersion of ceramic particles tend to block the dislocation motion and attribute to the dispersion strengthening mechanism which results in the improvement of various properties such as enhanced hardness, thermal conductivity, thermal shock resistance, electrical conductivity etc [2]. Properties of CMCs depend upon several factors like amount and the distribution of final phase formed as well as processing route applied for synthesis. A number of ceramic matrix composite systems have been synthesized using various synthesis routes as per the required area of application. D. Sarkar et.al. [3] synthesized the powder of Al_2O_3–ZrO_2 nanocomposite by using sol-gel synthesis route. Synthesized material was found to have their application in cutting tools and as prosthesis components due to their specific properties such as very good toughness, wear resistance and room temperature strength. Beitollahi et al. [4] investigated the synthesis and characterization of Al_2O_3–ZrO_2 nanocomposite powder by sucrose process. This process is a modified chemical route in which powder was prepared by using sucrose, poly vinyl alcohol (PVA) and metal nitrates followed by a post calcinations process. J. Kishan et.al. [5] have synthesized ZrO_2-Al_2O_3 nanocomposite powder by varying the amount of alumina 3-48%. They have utilized aqueous combustion synthesis route to achieve nano powder in their investigation. D. Goberman et al. [6] have worked on the synthesis of Al_2O_3–13wt. % TiO_2 plasma sprayed coatings obtained from nanocrystalline powders. Final microstructure was found to have bimodal distribution containing two different regions. One was having completely melt region and another one was having a microstructure with incomplete melted region. Ratio of complete melted region to the incomplete melted region can be controlled by a parameter known as critical plasma spray parameter (CPSP). Many properties such as mechanical strength, wear resistance, spalling resistance etc. have been improved due to this bimodal distribution of nanostructured Al_2O_3– 13wt. %TiO_2. C. Kaya et al. [7] worked on the synthesis of zirconia added mullite, bohemite and pure zirconia nano structure by hydrothermal synthesis route at comparatively low temperature. A porous morphology with near hexagonal particle shape was achieved in this investigation. The prepared nano structured materials have their application in fibre reinforced ceramic composites and ceramic fibre coating etc.

As mentioned above, there are various processing routes for the fabrication of ceramic matrix composites. Those routes include solid state route, sol-gel route, plasma spraying, laser synthesis, hydrothermal synthesis and co-precipitation route etc. Among all the routes, co-precipitation and hydrothermal routes are advanced routes to achieve the nano particles as well as homogenous microstructure of composite material. Further advancement has also been done by using polymer as a surfactant during synthesis. Various processing routes for the synthesis of CMCs are illustrated below.

2. Processing Techniques

A number of theories are available for synthesis of various ceramic matrix composites. Structural, thermal and mechanical properties depend on the processing route used for synthesis of ceramic matrix composite material. Therefore, selection of processing route plays an important role for achieving desired properties. A number of conventional and advanced processing routes have been used by various researchers for the fabrication of different kind of CMCs. Various processing routes are illustrated below.

2.1 Solid state route [8]

Solid state route is a conventional route to obtain different types of ceramic matrix composites. In this route, different oxide powders are mixed in a stoichiometric proportion to get the required composition. Prepared mixture of powders is kept for wet milling in a ball mill. This is done for few hours to make a homogenous mixture. Grinding media is generally used during mixing to achieve the fine and homogenous powder mixture and the ball to powder ratio and milling duration depends upon the required size of powder. Wet milling is preferred instead of dry milling for better homogeneity which is done in the presence of any liquid medium like propanol. Prepared mixed powder is dried and calcined at a required temperature for moisture and organic removal as well as phase transition. The synthesized powder is mixed with a very small amount of binder and compacted at certain pressure to obtain a desired shape. The prepared pellet is finally dried and then sintered at a high temperature with a soaking period of few hours for phase transformation, microstructure development and densification. Sintering is a process of heating at high temperature to provide bonding and strength to the material which is taken generally 70-90% of the melting temperature [9].

2.2 Sol-gel [10]

In this route, two or more precursor solutions are mixed together in a stoichiometric ratio. This solution is stabilized at room temperature along with continuous stirring. Ammonium hydroxide solution is added drop wise in this stirred solution. As a result of hydro-gel formation, the viscosity of solution increases continuously. pH is finally set to a value of 7-8 at a sufficiently high viscosity. Received gel is kept for ageing at room temperature for few hours. Formed precipitate is washed off by using distilled water. Washing is done for the removal of remaining acidic and basic ions, the filtered precipitate is dried at a temperature below 100°C for few hours. The dried gel is calcined at higher temperature with a hold period of few hours. Achieved powder is then investigated by using different characterization techniques.

2.3 Pechini method [11]

In this route, metal isopropoxide is mixed with nitric acid to form metal nitrate solution. Pechini method involves the formation of polybasic quelates between α-hydroxy carboxylic and metallic ions coming from metal nitrate nona-hydrate. pH of the final solution is maintained to a lower value of approximately 2. Polyesterification of the solution is done on heating below 100°C and stirring with a poly functional alcohol.

Further heating to a higher temperature at around 175°C produces a resin having high viscosity. This viscous resin is then transformed into a glassy gel which is rigid and transparent in nature. Attrition milling of this dehydrated gel is done for few hours to convert it in powder form. Sieving of the milled powder is done to achieve the required particle size of powder. The precursor oxide can be finally obtained by heating it at higher temperature.

2.4 Laser synthesis [12]

Laser synthesis includes the non-equilibrium vapor/plasma conditions created at the surface by intense laser pulse. In laser synthesis route, a powder mixture having the combination of different oxides is compacted in pellet form under certain load. The laser treatment of this compact is done by using continuous-action laser radiation (λ = 1064 nm). As a result of irradiation, concave channels (tracks) forms on the surfaces of specimens. Their formation is associated with high temperatures in the irradiation zone, due to which the sintering and melting takes place followed by solidification processes. These tracks were easily removed from the compacts. The lower sides of the tracks were cleaned from the loose slightly sintered powder. Phase formation also occurs during irradiation process. Synthesized products are investigated using different characterization techniques. Figure 1 shows an electron micrograph of cross section of channels formed after laser irradiation.

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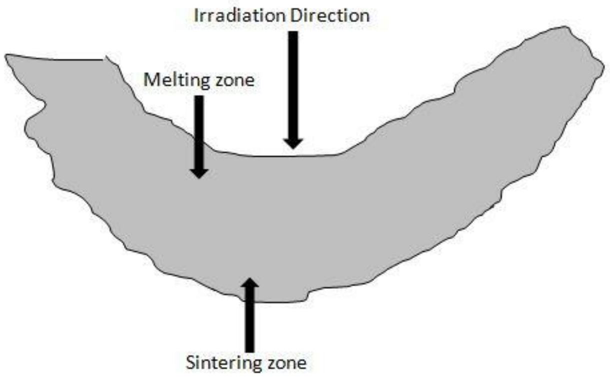

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Figure 1: Electron micrographs of cross section of channels formed after laser irradiation [10]

2.5 Melt synthesis [13]

Melt synthesis route includes the weighing of initial oxide powders and their mixing in ethanol medium. Mixture is dried followed by melting using an arc furnace. Small amount of graphite is added for radiation absorption during melting. A second melting is again done to ensure complete melting which is then cooled to room temperature. This is grounded to convert it in a powder form. Further investigations were done with the help of various characterization techniques.

2.6 Co-precipitation route [14]

In co-precipitation route, different metal salts are mixed homogenously and then precipitated with the help of any basic medium, generally using ammonia solution. Prepared precipitate is then washed to remove extra acid and basic ions. Drying, calcination and then grinding of the washed product is done to achieve powder. Co-precipitation can be done by using two different routes. First is direct precipitation route in which the precipitator is added and mixed homogenously to the salt solution and second is inverse precipitation route in which salt solution is poured and stirred into the precipitator to get precipitate. In second method, the formed precipitate is more finely dispersed than the precipitate by first method. Co-precipitation method offers following advantages. They are:

1. Simple and rapid preparation method.
2. Possibility of controlling particle size and composition easily.
3. Various possibilities to modify the particle surface state and overall homogeneity.
4. Smallest size particles with rounded shape can be obtained by using co-precipitation route.

2.7 Hydrothermal synthesis [15]

In hydrothermal route, fine powders can be synthesized by using initial precursor powder. These precursors are charged into a teflon-lined autoclave. The reactor is heated to a temperature in the range of approximately 100-150°C under some uniaxial pressure for different reaction times. Obtained powder is washed with distilled water to remove soluble part which is dried in hot air oven for few hours for complete removal of hygroscopic water. Sintering of the dried powder is done at a high temperature for few hours. The control of pH as well as solubility is an important aspect for hydrothermal synthesis. It is reported that the increase in pH value helps in reducing the particle size as well as de-agglomeration of the powder. Small size particles which have higher solubility, tends to form large crystals during sintering. This change in morphology helps in densification and thus the improvement in mechanical behavior occurs. Recent advancement is done by using a polymer as a surfactant during hydrothermal synthesis route. .

The combination of hydrothermal route with the use of surfactant helps in achieving following advantages:

- Particle size of the synthesized powder is in nano range (< 100 nm).
- Homogenous and fine grained microstructure of finally sintered product.
- Reduced synthesis temperature and short reaction time.
- Well crystallized solid solution which avoids further thermal treatments.

3. Recent Advancements

José ortiz - landeros et.al. [15] investigated the synthesis of lithium silicate by surfactant assisted hydrothermal crystallization method. Octylphenol ethylene oxide (TRITON X-114) was used as a surfactant in this route. Non-ionic surfactant (TRITON X-114) and LiOH reagents were dissolved in an ethylic alcohol aqueous solution. Tetraethyl orthosilicate (TEOS) in a stoichiometric amount was added drop by drop in the solution with continuous stirring for 15 minutes to make a homogenous mixture. The hydrothermal process was performed in a Teflon lined stainless steel autoclave vessel at a temperature of 100°C for 20 h under autogenously produced pressure. The estimated crystallite size was found to be approximately 14 nm based on XRD analysis. Whereas from the SEM micrograph, well defined spherical aggregates of size less than 3.5 µm were evident.

Xinzhang Zhou et al. [16] has synthesized zirconia–alumina–spinel nanoceramic composite by spark plasma sintering of plasma sprayed powders. This advanced method includes three steps:

1. Spray drying and plasma spraying which includes the mixing and ball milling of initial powder having submicron or nanosize range in water medium. Resultant slurry was spray dried to convert it in agglomerate form which is again converted into fine powder form. Plasma melting of the achieved powder was done at a 750A current and 65V voltage. Melted particles were quenched in a water bath.
2. Second step involves the ball milling and mixing with PVA to form a compact shape by using unidirectional load in a graphite die.
3. Final stage is an advanced sintering method at moderate pressure and low temperature which is known as spark plasma sintering. Spark plasma is generated between particles for heating purpose. A rapid heating is done with a simultaneous unidirectional pressure in a graphite die. Final cooling is done in vacuum which is then sent for further characterizations.

Zeinab Mosayebi et al. [17] investigated the surfactant assisted co-precipitation route for synthesis of magnesium aluminate spinel. Pluronic P123 was used as surfactant in this investigation. Surfactant was dissolved in water and stirred to make it completely dissolved. Magnesium nitrate hexahydrate and aluminum nitrate were dissolved in the P123 solution. Drop wise addition of aqueous ammonia is done for the formation of precipitate. Mechanical stirring of the slurry was done for approximately 30 minutes. The prepared slurry was filtered and washed with deionized water to remove surfactant completely and the final product was dried at a temperature of 80°C for 24 hrs. Grinding of this dried product was done followed by calcinations at 600°C, 700°C and 800°C for 4 h. Particle size of the achieved powder by using this route was in nano size range (9-10 nm).

The present research work has been done on the synthesis of Al_2TiO_5 composite nano powder by surfactant assisted hydrothermal route. Figure 2 shows the flow diagram of synthesis route by surfactant assisted hydrothermal route used in the present work. The prepared powder is dried in hot air oven and then ground in fine size using agate mortar for 20-25 mins. Plot of DSC/TGA is done to analyze the thermal behavior of prepared powder. The DSC/TGA has been carried out using STA449C/4/MFC/G, Netzsch, Germany at a rate of 10^{o}C/min in argon atmosphere. XRD of the calcined powders was done using Rigaku Desktop Miniflex II X-ray diffractometer, employing Cu Kα radiation and Ni-filter whereas microstructure was recorded using FEI NOVANANO SEM 450 field emission scanning electron microscope.

Figure 3 shows the DSC/TGA plot for Alumina-Titania powder. There are two endothermic and one exothermic peak shown in the plot.

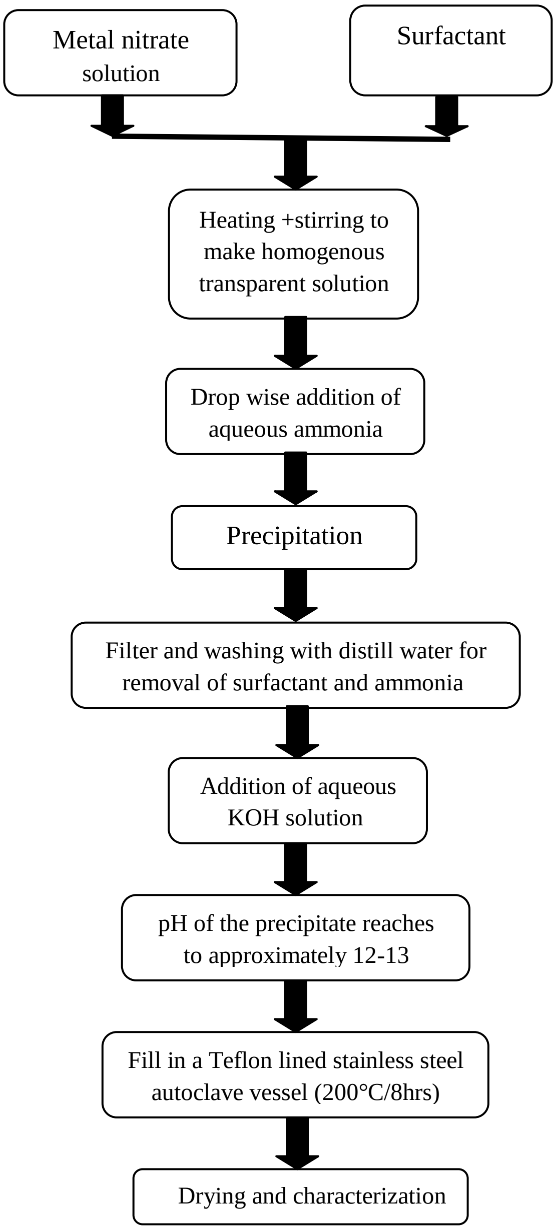

Figure 2: Flow diagram of synthesis route by surfactant assisted hydrothermal route

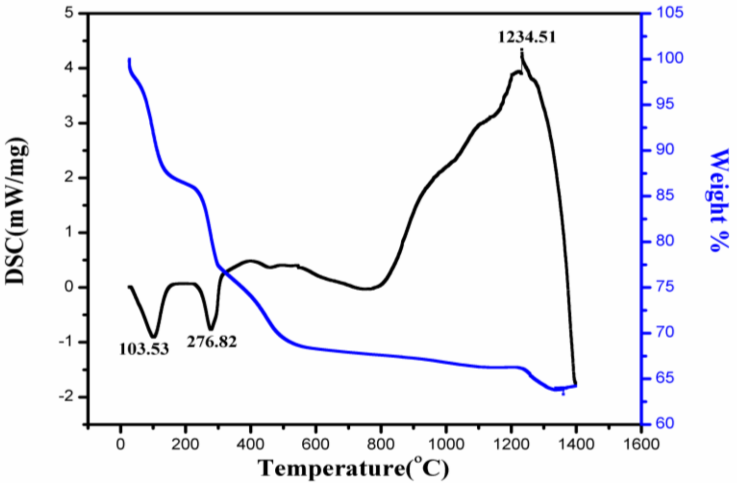

Figure 3: DSC/TGA plot for synthesized powder by hydrothermal route

Two endothermic plots are evident at 103.53°C and 276.82°C with total weight loss of 8% and 13% respectively. A next exothermic peak is visible at a temperature of 1234.51°C with a weight loss of 15%. Peak at 103.53°C is related to the decomposition of adsorbed water whereas peak at 276.82°C corresponds to early decomposition of gibbsite into γ- alumina phase [11]. Temperature around 1300°C is related to the formation of Aluminium titanate [1].

Fig. 4 shows the X-ray pattern of synthesized powder prepared by hydrothermal treatment. Alumina and Titania are reacting with each other to form a new phase Tialite (Aluminium Titanate) during hydrothermal treatment [18]. Phase formation of Tialite (Al_2TiO_5) is evident from the figure 4. The other two phases are alumina (Al_2O_3) and rutile (TiO_2). Fig. 5 shows the FESEM micrograph of Al_2O_3-TiO_2 composite powder synthesized by surfactant assisted hydrothermal treatment at a magnification of (a) 10000X and (b) 100000X. From the micrograph, it is easily evident that the synthesized powder is in the range of nanometers [19-20]. The achieved powder is rounded in shape, non-agglomerated and well distributed.

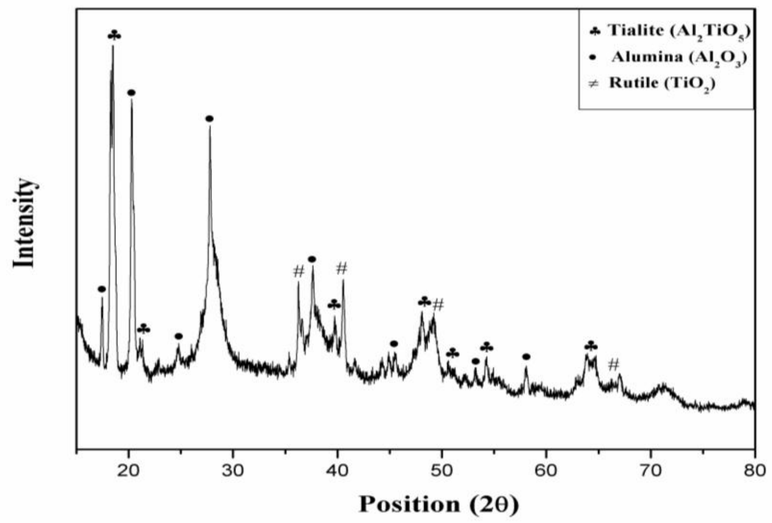

Figure 4: X-ray diffraction pattern of synthesized powder by hydrothermal route

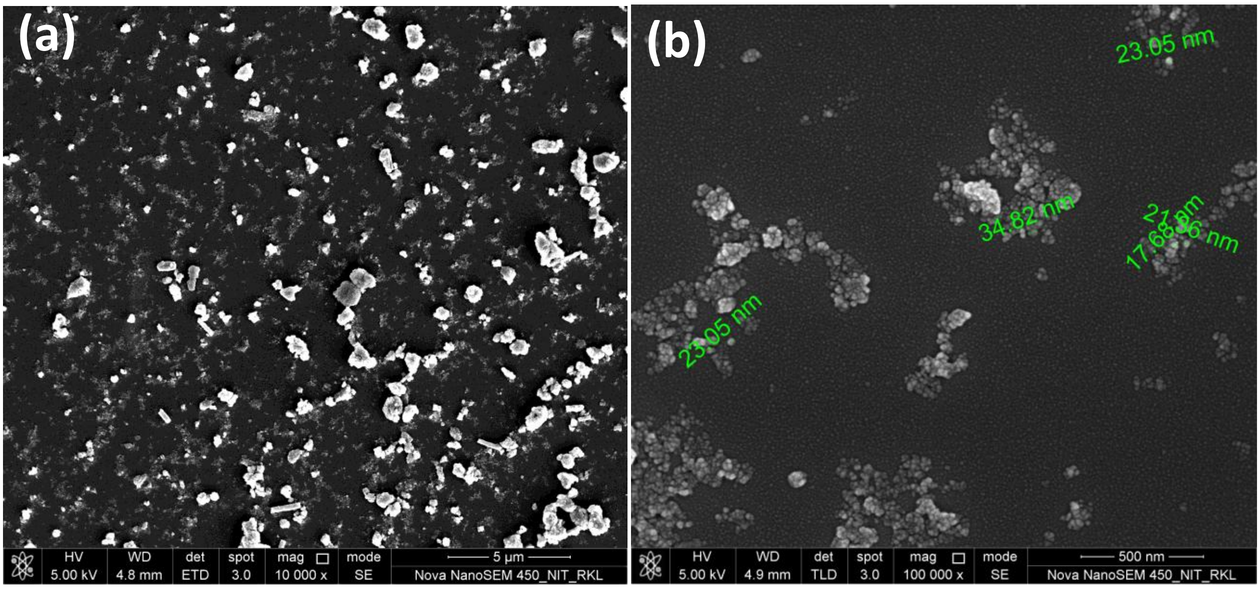

Figure 5: FESEM micrograph of synthesized powder by hydrothermal route (a) 10000X (b) 100000X

4. Industrial Applications

The superior properties of ceramic matrix composites have led to identify a number of industrial applications for efficient working. There are some applications of various ceramic matrix composites as follows:

- Aeronautical and automotive purposes
- Orthopedic and dental implants
- Petroleum hydro-treatments

- Potential adsorbent in chemical warfare
- Solar cells
- Humidity sensors
- Electronic applications
- Electrolyte for solid oxide fuel cell
- High speed cutting tool applications
- Structures for gas turbine engines
- Electrical and thermal insulators

5. Conclusions

A systematic study on various processing routes to synthesize ceramic matrix composites is done in the present investigation. The reinforcement of a secondary ceramic phase in some ceramic matrix has been found beneficial to improve the mechanical and thermal behavior of composite. There are some important conclusions drawn in the present investigation which are as follows:

1. A number of processing routes have been utilized for the synthesis of ceramic matrix composites for several years.
2. Among them, co-precipitation and hydrothermal routes are widely used to synthesize CMCs. A lot of work has been done on the conventional routes but there are a few reports on surfactant assisted routes to achieve nanocomposites.
3. Surfactant assisted co-precipitation and hydrothermal routes have achieved special attention due to the better achieved morphology of final synthesized product.
4. Particle size achieved by both of the route as mentioned above is in nano size range (<100nm). Synthesized nanocomposite has gained better thermal, mechanical and structural properties for various applications.

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(2017) ; http://www.jmaterenvironsci.com