

Rapid growth approaches from nano-Mg(OH)₂ to bulk materials and their application in the environment

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Introduction

In recent years, nano-adsorbents have emerged as a fast-developing and effective treatment of pollutants [1, 2], especially suited for removing low-concentration pollutants in wastewater [3, 4]. Due to their small size, they have a large surface area and a great number of active sites, which lead to a strong adsorption capacity for the pollutants. However, after adsorbing pollutants they become so-called nanowastes, which are harmful substances for the environment [5, 6]. Nowadays, burial or storage is the general method for treatment of the loaded nano-adsorbents [7, 8]. Although the aim of this kind of method is permanent sequestration of the contaminants, there is still a very high possibility that the contaminants would ultimately leach from the solid wastes into groundwater again due to the poorly known stability of the resulting material.

On the other hand, some traditional industrial sludges have been gradually recognized as nanowastes with adsorbed heavy metals or organic pollutants. For instance, more than 800 kilotons of Mg(OH)₂-containing nanowastes are generated from chlor-alkali and chlorate plants in China annually [9]. Sometimes these nanowastes have tightly bound toxic heavy metals. The Mg(OH)₂ nanowastes from the sodium and potassium chlorate industries, for example, typically have adsorbed carcinogenic Cr(VI), because sodium dichromate is commonly used in chlorate plant to maintain high current efficiency [10, 11]. The Mg(OH)₂ nanoparticles have such a high surface-adsorption affinity for the toxic Cr(VI) that it is difficult to remove the Cr(VI) ions from the solid surface through traditional methods such as washing, filtration and centrifugation. The conventional treatment of this Cr(VI)-containing sludge is through landfill or storage after reduction of toxic Cr(VI) to less toxic Cr(III) [12, 13], whereas it may probably cause secondary pollution because of the risks that the Cr(III) would be reoxidized into Cr(VI) and the chromium would be accumulated through the food chain.

Heavy metals, such as hexavalent chromium, uranium and arsenic, have toxic and mutagenic effects even at very low concentrations [14, 15]. As priority pollutants, they are not biodegradable and tend to accumulate in living organisms, causing persistent environmental contamination and becoming a severe public health problem. Therefore, the ideal strategy for disposing either heavy metals loaded nano-adsorbents or heavy metals-containing nanowastes is to eventually recycle and reuse the heavy metals.

From this point of view, we proposed a new approach for treatment of heavy metals-containing nanowastes by converting the nanowastes into a nontoxic bulk material and separating the concentrated heavy metals solution for reuse. This article takes Cr(VI) adsorbed Mg(OH)₂ nanowastes for example and reviews the approaches of rapid growth from nano-Mg(OH)₂ to bulk material

in the study of our group, and provides a recycling strategy of Mg(OH)₂ nano-adsorbents for dealing with aqueous solution of low-concentration Cr(VI).

Treatment of Cr(VI)-adsorbed nanowaste by mineralizer

The Cr(VI)-containing Mg(OH)₂ nanowaste was generated from chlorate industry during the brine purification step. This nanowaste is composed of about 50% water and more than 2000 mg/kg of Cr(VI), and the main solid composition is 20-nm Mg(OH)₂ and 100-nm CaCO₃. Figure 1 shows the photos of a settling tank in the chlorate plant and the Cr(VI)-containing nanowaste after pressure filtration. The nanoparticles in the tank are very difficult to settle and after pressure filtration the nanowaste became viscous and tightly adsorbed with toxic Cr(VI).

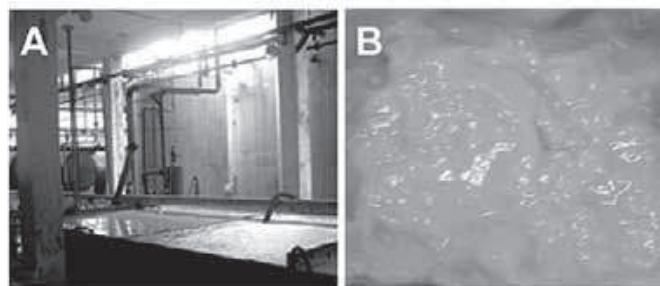


Fig. 1. A) the settling tank in chlorate plant, B) the Cr(VI)-containing nanowaste after pressure filtration

Based on our previous investigation on the thermodynamics and kinetics of nano-crystal growth [16 ÷ 19], a strategy for disposing this Mg(OH)₂ nanowaste was proposed as follows. We seek to convert the Mg(OH)₂ nanoparticles into bulk material for the separation and recycling of the Cr(VI) from the solid. The NaHCO₃ + Na₂CO₃ or NaHCO₃ was used as the mineralizer to accelerate the rapid growth of nano-crystals.

Figure 2 shows that the original nanowaste contained a lot of Mg(OH)₂ nanoparticles, which were difficult to settle in the solution (Fig. 2 A and D). After coarsening in water (without mineralizer), there was no obvious change in the size and settling properties of the solid particles (Fig. 2 B and E). This suggests that the Mg(OH)₂ phase could remain stable on a nanoscale for a long time under naturally occurring conditions. However, when the nanowaste was coarsened with mineralizer, the solid particles settled down to the bottom of the beaker in only 20 min. The SEM image revealed that the nanoparticles had already been converted into micron-sized crystals (Fig. 2 C and F). The leaching test of Cr(VI) proved that only the samples after the treatment with mineralizer could meet the standard of environmental protection technical specifications for treatment of chromium residues in China (HJ/T 301-2007, 0.5 mg/l).

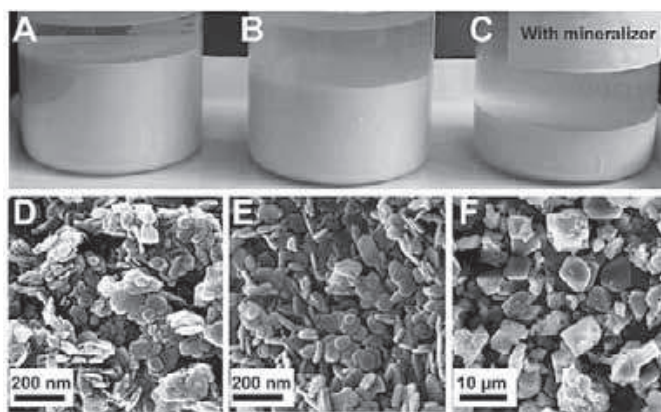


Fig. 2. The settling test and SEM images of the samples: **A and D)** the original $\text{Mg}(\text{OH})_2$ nanowaste; **B and E)** the sample coarsened in pure water (without mineralizer) at 180°C for 24 hours; **C and F)** the sample coarsened in mineralizer **B** (NaHCO_3 1.5M) at 180°C for 24 hours. The solid/liquid ratio used in the settle ability test was 500 g/5000 ml, with a settling time of 3 hours. The use of mineralizer **A** ($\text{NaHCO}_3 + \text{Na}_2\text{CO}_3$) was reported in previous study [20]

The mechanism of Cr(VI) removal during the whole process of coarsening $\text{Mg}(\text{OH})_2$ nanowaste by the mineralizer is shown in Figure 3. The separation of Cr(VI) from the solid includes two steps: i) desorption of Cr(VI) from the solid surface due to the aggregation of $\text{Mg}_5(\text{CO}_3)_4(\text{OH})_2 \cdot 4\text{H}_2\text{O}$ nanolamellas, which was transformed from $\text{Mg}(\text{OH})_2$ nanoplates under the effect of the mineralizer. ii) efficient separation of the concentrated Cr(VI) solution from the solid particles due to the rapid crystal growth, which displayed an obvious increase of settling velocity [20].

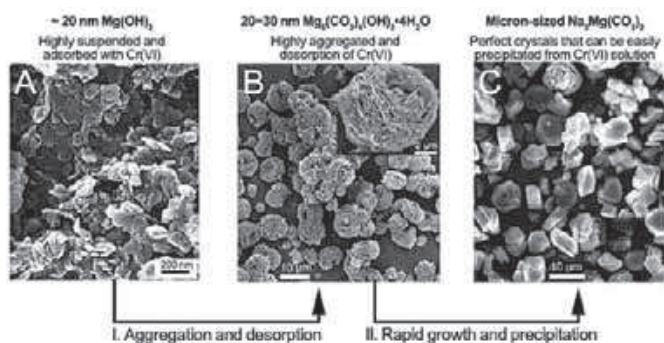


Fig. 3. The mechanism of Cr(VI) removal during coarsening $\text{Mg}(\text{OH})_2$ nanowaste by mineralizer

In the first step, the desorption of Cr(VI) and aggregation of $\text{Mg}_5(\text{CO}_3)_4(\text{OH})_2 \cdot 4\text{H}_2\text{O}$ nanolamellas were probably due to the change of the surface charge state. The initial pH of the nanowaste is around 9, which is lower than the isoelectric point of $\text{Mg}(\text{OH})_2$ (approx. 11.9), therefore the surface of $\text{Mg}(\text{OH})_2$ nanoplate is positively charged. The positive charge can both facilitate the adsorption of Cr(VI) anions ($\text{Cr}_2\text{O}_7^{2-}$ or CrO_4^{2-}) on the surface of $\text{Mg}(\text{OH})_2$ nanoplates and prevent the aggregation of the nanophase

(Fig. 3A). During the mineralizer treatment, the $\text{Mg}(\text{OH})_2$ nanoplate can react rapidly with HCO_3^- ions by surface adsorption, which results in the formation of $\text{Mg}_5(\text{CO}_3)_4(\text{OH})_2 \cdot 4\text{H}_2\text{O}$ nanolamellas with a similar size. Meanwhile, the decrease of surface charges in this step altered the aggregation state of lamellar $\text{Mg}_5(\text{CO}_3)_4(\text{OH})_2 \cdot 4\text{H}_2\text{O}$ as compared to that of $\text{Mg}(\text{OH})_2$ nanoplate, leading to the desorption of Cr(VI) anions (Fig. 3B).

In the second step, the growth rate of $\text{Na}_2\text{Mg}(\text{CO}_3)_2$ crystals (several micrometers per hour) is extremely high compared to the materials with similar solubility, such as ZnS in 4 M NaOH solution (0.02 nm/h via oriented attachment growth, or 0.0026 nm/h via ostwald ripening growth). A collective phase-transformation mechanism may

be responsible for the rapid growth of nanocrystal from aggregated $\text{Mg}_5(\text{CO}_3)_4(\text{OH})_2 \cdot 4\text{H}_2\text{O}$ nanolamellas to micron-sized $\text{Na}_2\text{Mg}(\text{CO}_3)_2$ crystals. Currently, further research about rapid growth of $\text{Na}_2\text{Mg}(\text{CO}_3)_2$ crystals based on aggregation is being conducted.

After the optimization of treating conditions in industry, the pilotscale tests were carried out with 1 tonne of Cr(VI)-containing nanowaste [21, 22]. More than 97% of Cr(VI) could be effectively removed by this method with a low Cr(VI) leaching of less than 0.5 mg/l. Moreover, the concentrated Cr(VI) solution could be reused in the chlorate plant, and the detoxified solid was attempted to be recycled in ceramic and metal coatings.

In order to reduce the cost of treatment and save energy, treatment method of Cr(VI)-containing nanowaste at room temperature was developed. Figure 4 compares the photos and micro-morphologies of the original nanowaste and the sample treated at room temperature. It was found that the rapid growth approach from $\text{Mg}(\text{OH})_2$ nanoparticles to bulk material at room temperature was different from that at the high temperature. In this method, the $\text{Mg}(\text{OH})_2$ nanoparticles was converted into $\text{MgCO}_3 \cdot 3\text{H}_2\text{O}$ (nesquehonite) crystal clusters whose size were hundreds of microns, leading to the desorption of Cr(VI) from solid surface. Further study is being carried out for elucidate the rapid growth mechanism at room temperature.

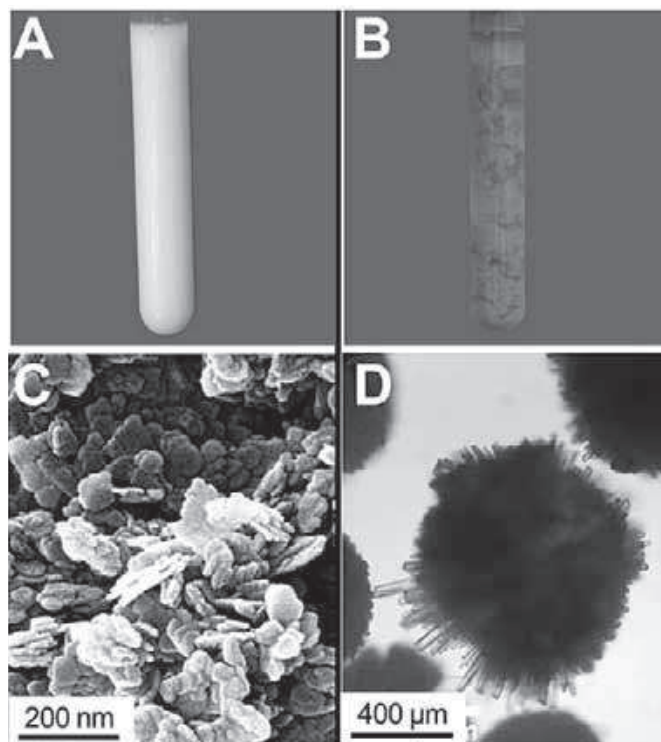


Fig. 4. The photos and micro-morphologies of the samples: **A)** the photo of original $\text{Mg}(\text{OH})_2$ nanowaste, **B)** the photo of the sample treated in mineralizer **B** at room temperature for 48 h, **C)** the SEM image of the original sample and **D)** the optical micrograph of the treated sample

Recycling $\text{Mg}(\text{OH})_2$ nano-adsorbent for preconcentration of Cr(VI)

Cr(VI) is of particular concern due to its highly toxic, mutagenic and carcinogenic effect even at low concentration. Various techniques have been employed for the removal of Cr(VI) from wastewater. The reduction and precipitation treatment [23, 24] is the most mature and economical technology for the wastewater containing tens to hundreds mg/l of Cr(VI). When Cr(VI) is at low concentration, i.e. less than 10 mg/l, a much larger amount of dosage is needed, producing a great amount of sludge that requires further treatment. Other methods, such as ion exchange [25], electrolysis [26] and membrane filtration [27], although usually effective, require a high capital investment and

the cost of treatment is high. The nano-adsorbent seems to be the most promising technique for dealing with low concentration of Cr(VI) due to low cost, ease of operation and efficiency [28].

As a low-cost, non-toxic and environmentally friendly nano-adsorbent, Mg(OH)₂ has been widely used in neutralization of acidic wastewater [29], removal of heavy metals [30], color removal from dye-containing wastewater [31], removal of phosphate and ammonium from municipal wastewater [32] and flue gas desulfurization [33]. In addition to removing heavy metal cations by co-precipitation, Mg(OH)₂ also has the potential for the removal of highly toxic anionic pollutants, such as hexavalent chromium (Cr₂O₇²⁻, HCrO₄⁻ and CrO₄²⁻) and arsenic (AsO₄³⁻ and AsO₃³⁻), due to the positively charged surface. Over the past decade, global consumption of Mg(OH)₂ in wastewater treatment has reached more than 48,000 tons per year [33], which created great amounts of Mg(OH)₂ sludge. Similar to loaded Mg(OH)₂ nano-adsorbent, the other nano-adsorbents become harmful substances as mentioned above. Therefore, for large-scale application of nano-adsorbents in wastewater treatment, the recycling of nano-adsorbents should be considered.

In view of the above, the ideal method for treating low concentration of Cr(VI) is using recyclable nano-adsorbents to pre-concentrate Cr(VI) for recovery or reuse. A common problem for recycling nano-adsorbents is that the initial surface state and the morphology of the nano-adsorbents may gradually disappear after several cycles due to the inherent thermodynamic instability of the nanophase [34], i.e. the growth and aggregation of nanoparticles. The most effective solution for this problem is to create a reversible transformation between the nano-adsorbent and bulk material.

Our study showed that the nano-Mg(OH)₂ could be changed into bulk MgCO₃·3H₂O under the effect of NaHCO₃ at room temperature. Hence, it is easy to design a reversible route between nano-Mg(OH)₂ and bulk-MgCO₃·3H₂O [35], as shown in Figure 5. In order not to create other chemical byproducts during the treatment, the CO₂ was used instead of the mineralizer (NaHCO₃). Three main steps were included in the recycling of the Mg(OH)₂ nano-adsorbent: (I) using Mg(OH)₂ nano-adsorbent to adsorb low concentration of Cr(VI) from wastewater, (II) transforming Cr(VI)-loaded Mg(OH)₂ nanoplates into bulk MgCO₃·3H₂O via rapid phase transformation and crystal growth induced by CO₂, (III) turning the bulk MgCO₃·3H₂O back into the same Mg(OH)₂ nanoplates via calcination and hydration treatment.

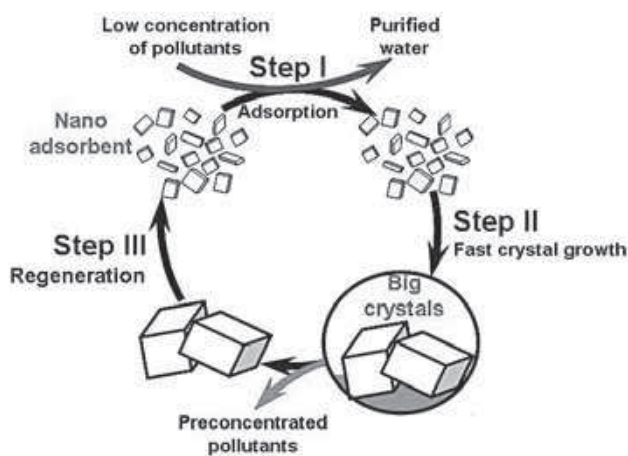


Fig. 5. The scheme of experimental design for recycling Mg(OH)₂ nano-adsorbent during treating of low concentration of Cr(VI)

In the first step, nano-Mg(OH)₂ was prepared by putting the heated MgO (600 °C) directly into wastewater with low concentration of Cr(VI), which combines the adsorbent generation and pollutant

adsorption into one step. The SEM image shows that the synthesized Mg(OH)₂ was in the shape of nanoplates (Fig. 6A). After rapid growth of nanocrystals induced by CO₂, the Mg(OH)₂ nanoplates turned into micron-sized MgCO₃·3H₂O rods (Fig. 6B). The MgO kept the same size and morphology after calcination (Fig. 6C), but the Mg(OH)₂ nanoplates were regenerated when the heated MgO was hydrated (Fig. 6D). As expected, the results showed that the regenerated Mg(OH)₂ nanoplates still had good adsorption of Cr(VI). During the recycling of Mg(OH)₂ nano-adsorbent, Cr(VI) could be enriched more than 40 times.

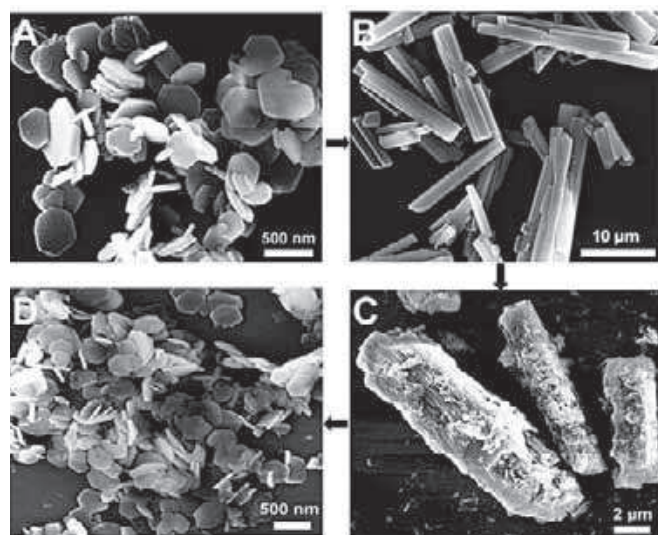


Fig. 6. The SEM images of the samples: A) synthesized Mg(OH)₂ nano-adsorbent, B) bulk MgCO₃·3H₂O, C) MgO from calcined MgCO₃·3H₂O, and D) regenerated nano-Mg(OH)₂

The rapid growth mechanism from nano-Mg(OH)₂ to bulk MgCO₃·3H₂O was further studied. The preliminary data indicated that the growth of nanocrystals may involve a two-step mechanism, in which Mg(OH)₂ lamellas first aggregate and form a rod-like agglomerate followed by *in-situ* conversion of aggregated nanoparticles to bulk crystals. More in-depth investigations are still in progress.

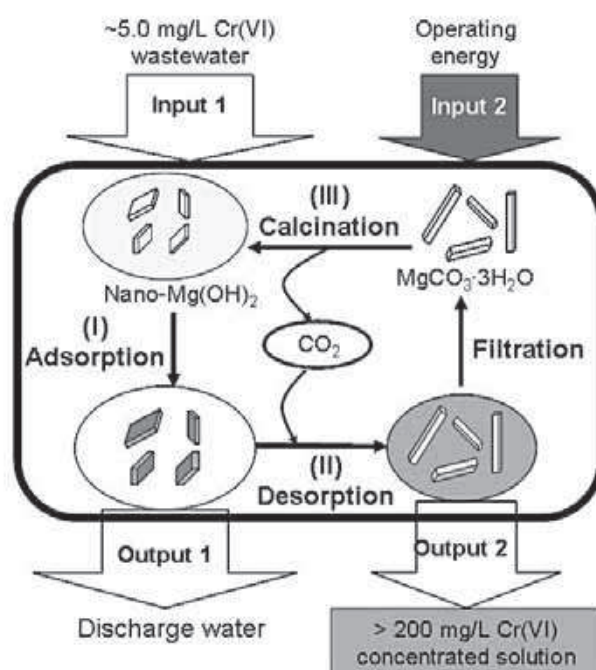


Fig. 7. Summary of nano-Mg(OH)₂ recycling system for pre-concentration of Cr(VI)

Because the amount of consumed CO_2 during the desorption process (step II) was equal to the amount of CO_2 released during the regeneration process (step III), recycling nano- $\text{Mg}(\text{OH})_2$ did neither require any external chemical reagents nor generate any chemical byproducts. The recycling loop of $\text{Mg}(\text{OH})_2$ nano-adsorbent combined with the CO_2 medium can be considered as a "black box", where the input substance was low-concentration wastewater, the outputs were purified water and enriched $\text{Cr}(\text{VI})$ solutions, as illustrated in Figure 7. Except for the operating energy, no extra chemical agents were required or produced. Therefore, the carbonation strategy for recycling $\text{Mg}(\text{OH})_2$ nano-adsorbent was eco-friendly from the environmental point of view. This recycling strategy of $\text{Mg}(\text{OH})_2$ nano-adsorbent can also be used for treating low concentration of other highly toxic anionic pollutants, such as AsO_4^{3-} or $[\text{UO}_2(\text{CO}_3)_3]^{4-}$. Moreover, this strategy of fast nanocrystal growth can also be applied to dispose other toxic nanowastes, such as heavy metal-contained aluminum hydroxide sludge and electroplating sludge [36].

Summary and conclusions

This article reviews the study of our group on the rapid growth approaches from nano- $\text{Mg}(\text{OH})_2$ to bulk material and their environmental applications. The first application of the rapid growth of nanocrystal is in the treatment of pollutants-adsorbed nanowastes. The treatment of $\text{Cr}(\text{VI})$ adsorbed $\text{Mg}(\text{OH})_2$ nanowaste exemplified the transformation of the $\text{Mg}(\text{OH})_2$ nanoplates into nontoxic bulk materials, i.e. micron-sized $\text{Na}_2\text{Mg}(\text{CO}_3)_2$ or $\text{MgCO}_3 \cdot 3\text{H}_2\text{O}$ crystals, and consequently the desorption and complete separation of heavy metal $\text{Cr}(\text{VI})$ from the solid. The second application is developing a recycling strategy of $\text{Mg}(\text{OH})_2$ nano-adsorbent for treating the wastewater with low concentration of highly toxic anionic pollutants, such as CrO_4^{2-} , AsO_4^{3-} or $[\text{UO}_2(\text{CO}_3)_3]^{4-}$. A carbonation method was successfully applied to the recycling of $\text{Mg}(\text{OH})_2$ nano-adsorbent based on the reversible route between the nano- $\text{Mg}(\text{OH})_2$ and bulk $\text{MgCO}_3 \cdot 3\text{H}_2\text{O}$ for preconcentration of the $\text{Cr}(\text{VI})$. The mechanistic study indicated that the fast growth from nano- $\text{Mg}(\text{OH})_2$ to bulk $\text{MgCO}_3 \cdot 3\text{H}_2\text{O}$ underwent an *in-situ* conversion of aggregated nanoparticles to bulk crystals. This strategy of fast nanocrystal growth provides a good example for dealing with toxic nanowastes or recycling nano-adsorbents.

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The scientific program will include plenary invited lectures, oral communications, poster presentations, satellite workshops, as well as young researchers platform.

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The scientific program covers following areas:

- compounds associated with nutritional and sensory quality of foods: reactions and interactions under storage/processing conditions; reaction pathways, predictive modelling, the impact of novel technologies on physical-chemical changes; major food constituents (proteins, carbohydrates, lipids); the Maillard reaction and AGEs; flavours, texture and other quality/safety markers; minerals, vitamins and other constituents
- biologically-active constituents of foods and food raw materials: factors affecting their formation and changes during post-harvest handling; compounds, ingredients and formulations with nutritional and health benefit; toxic/anti-nutritive compounds
- strategies to improve food quality and safety: trends in food production, processing, packaging, and distribution; formation and mitigation of processing contaminants; products from organic farming; benefit/risk perception and communication in the food chain
- chemistry behind novel foods: composition, authentication, traceability of origin, stability, and shelf-life testing; nanotechnology in food processing; functional foods including prebiotics and probiotics; nutraceuticals and ingredients for food and medical uses.

Web Site: <http://www.crf2012.eu/>