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Review article

Approaches to synthesize MgO nanostructures for diverse applications

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ABSTRACT

Magnesium oxide remained interesting from long time for several important phenomena like; defect induced magnetism, spin electron reflectivity, broad laser emission etc. Moreover, nanostructures of this material exhibited suitability for different kinds of applications ranging from wastewater treatment to spintronics depending upon their shape and size. In this way, researchers had grown nanostructures in the form of nanoparticles, thin films, nanotubes, nanowalls, nanobelts. Though nanoparticles and thin films are well known form of nanostructures and wide variety of synthesis approaches are available, however, limited methodology for other nanostructures are available. In order to grow these nanostructures in an optimized way an understanding of these methods is essential. Thus, this review article depicts an overview of various approaches for design of different kinds of nanostructures.

1. Introduction

With the discovery of carbon nanotubes [1], development of nanostructures of different materials have evoked much attention for their fabrication, characterization and applications in various fields [2, 3, 4]. The unique properties of these nanostructures are not only intriguing for investigation of underlying phenomena but also opened several dimensions for their utilization in different technologies [5, 6, 7, 8]. Nanostructured oxides of different shape gain significant attentions in last two decades. Apart from nanoparticles, and thin films, most desirable form of nanostructures are nanocubes, nanorod and nanoflowers. These kinds of nanostructures and their applications are studied for numerous oxide systems. Depending upon the morphology, these form of nanostructures exhibit superiority over the nanoparticles and thin films. Hence, this review article is motivated to understand the growth behavior of different kind of nanostructures by considering MgO as a prototype system.

1.1. Nanostructures

Nanostructures represent a wide category of materials having dimensions less than 100 nm, which includes nanoparticles, thin films, nanotubes and nanorods [9, 10, 11]. Figure 1 depicts various category of nanostructures. It is clear from the figure that nanostructures have at least one dimension of the order of nm.

Due to small dimension of nanostructures, some exciting phenomena like quantum confinement [12, 13, 14, 15] and phonon confinement in these nanostructures take place [16, 17, 18]. This causes modulation in the optical behavior of nanostructures of various materials like Au, Ag [19], ZnO [20] and similar other systems [21, 22]. Thus, these nanostructures exhibit completely different behavior compared to bulk counterpart. The modulated behavior of nanostructures is not limited to the optical properties but also reported for magnetic [23,24], electrical [25,26] and thermal properties [27,28]. For example, paramagnetic zinc ferrite (with large particle size)

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Figure 1. Category of nanostructures: (a) nanoparticles, (b) thin film, (c) nanorod, (d) nanoplate, (e) nanotube and (f) tetrapod.



Figure 2. Some interesting phenomena observed in MgO nanostructures.

transforms into ferromagnetic when synthesized in the form of nanoparticles [29,30] or thin films [31]. Nanoparticles and thin films of several non-magnetic oxides are also reported to exhibit ferromagnetic ordering [32, 33, 34].

1.2. Specific phenomena observed in MgO nanostructures

Among the other non-magnetic oxides, magnesium oxide has become interesting from last two decades due to existence of several phenomena in it's nanostructures. Figure 2 summarizes phenomena that usually observed in the nanostructures of this material.

Among the various non-magnetic oxide system, MgO is a technologically important material because of its simple crystal structure and complete absence of *d* orbital electrons, which persists way of understanding several physical [35, 36, 37] and chemical behavior [38, 39, 40, 41].

Bulk MgO is highly insulating material with a very high optical bandgap of \sim 7.6 eV [42], however, nanostructures of this material exhibit modified optical band-gap. The optical band-gap of MgO nanoparticles of size 7 nm is 2.8 eV [43]. One-dimensional nanostructure of this material shows an optical band-gap of 3.2 eV [44]. Similar value of optical band-gap is observed for MgO nanocubes [45].

Though, bulk MgO and thin films exhibit dielectric constant of 10 [46, 47, 48], but this material attains high value of dielectric constant depending upon the morphology of nanostructure [49, 50, 51].

Spin-dependent electron reflection is reported in MgO thin films grown on Fe substrate. The electron reflectivity exhibits quantum interference from which two MgO energy bands with Δ_1 symmetry were determined in the experiment [52, 53, 54].

Resistive switching is observed in MgO based structures [55,56]. Ferromagnetism combining with multilevel switching characteristics is also reported in MgO capacitor [57,58]. Room-temperature broadband laser emission in the near ultraviolet to the blue-green spectral range was observed in MgO microcrystals obtained through a solid phase reaction between SiO and Mg at 450 °C in an Ar atmosphere [59,60]. Luminescence is another phenomenon observed in nanocrystals of MgO [61, 62, 63]. Other categories of luminescence like photoluminescence [64,65], electroluminescence [66], radioluminescence [67] and thermoluminescence [68] are also reported in MgO nanostructures.

It is observed that the presence of these phenomena do not only exist at reduced dimensions, but also reflect when defects accumulate in this material. For example; thin films and nanoparticles of magnetism oxide show magnetism associated with surface and extended defects [69, 70, 71]. Moreover, luminescent behavior of nanostructured MgO is tailored by controlling the defect states in the material [72, 73, 74].

Cataluminescence is another important phenomenon that occurs in MgO based nanostructures and persists a way for utilization as gas sensor [75, 76, 77].

1.3. Application of MgO nanostructures

Nanostructures of MgO are not only known for the observation of numerous phenomena as elaborated in section 1.2 but also because of significant enhancement of their applications in a variety of fields. MgO nanostructures are used as ultra-violet (UV) photodetector [78]. The photocatalytic activity of MgO nanoparticles are being utilized for degradation of methyl orange and methylene blue dyes under UV light irradiation [79].



Figure 3. The schematic is depicting the importance of MgO nanostructures in numerous field.

It is used as protective layer in alternating current (AC) plasma display [80] and as a substrate for chemical reactions [81, 82, 83]. The catalytic [84,85] and antibacterial activities [86,87] of MgO have attracted a significant scientific interest in recent years.

MgO also finds applications in pharmaceutical preparation, when mixed with drug that is unstable in acid, demonstrates high stabilizing effects on the drug [88]. Moreover, shape of nanostructures plays important role for diverse therapeutic applications [89]. It is used as an antiseptic compound while mixed with iodine in ratio of 97:3 [90]. The nanoparticles of MgO exhibit potential for environmental and biomedical applications [91, 92, 93]. MgO nanoparticles also show toxicity (17%) towards the cancer cell, which enables their possible utilization for cancer treatment [94, 95, 96].

MgO is also utilized in wastewater treatment for removal of chloride ions [97] as well as precipitation of phosphors and nitrogen [98, 99]. Porous MgO microrods are effective for removal of Pb and Cr [100,101]. The capacity of removal of similar heavy metal ions is influenced by morphology of MgO nanostructures [102, 103, 104, 105]. Incorporation of MgO nanoparticles helps to carbon, graphene oxide and Si to improve it's efficiency for CO₂ capture [106, 107, 108]. CO₂ capture efficiency is influenced by the shape of nanostructure [109].

It is being utilized for electrical insulation [110,111]. Magnetic property of this material make enable its use in low-field magnetic sensor [70]. Nevertheless, utilization of MgO as a barrier layer in magnetic tunnel junction has increased its importance in the field of spintronics [112,113].

Surface of this material has become promising for splitting of organic molecules like ethanol and methanol, which is helpful for production of H₂ [114,115]. Prediction of water splitting on MgO thin film/nano-particle surface is also made [116,117], which upon realization will be a boon for the production of clean energy.



Figure 4. SEM Image of MgO nanoparticles synthesized from magnesium nitrate. Adapted from Ref. [133] with permission from The Royal Society of Chemistry.



Figure 5. The mechanism of MgO formation. Reprinted from Ultrasonics Sonochemistry, 17/2, Mohammad Amin Alavi, Ali Morsali, Syntheses and characterization of Mg(OH)₂ and MgO nanostructures by ultrasonic method, 441–446, Copyright (2020), with permission from Elsevier [136].

MgO coating on cathode materials is effective to enhance electrochemical performance of rechargeable batteries [118, 119, 120]. Similarly, it's coating on selected anode materials for various rechargeable batteries is found effective to improve the performance [121,122]. In addition to above applications, this material is widely studied for understanding behaviors of materials under planetary conditions [123, 124].

Figure 3 depicts the important field where nanostructures of this material play important role.

Thus, this material exhibits interesting behavior and diverse applications depending on nature of nanostructures. The deposition or synthesis methods play important role in determining the nature of nanostructures [125, 126, 127]. Thus, there is need to get profound understanding of these methods. Hence, this review article covers the approaches to design various types of nanostructures of MgO for their utilization in diverse applications.

2. Nanoparticles

Nanoparticles are synthesized using various approaches, here; some processes are depicted in the following paragraphs. The objective of these synthesis methods is to get control over size, size distribution and shape of nanoparticles [128, 129, 130].

2.1. Sol-gel process

This method involves any water-soluble salt of magnesium. The solution of this slat is mixed with appropriate organic host and is converted to gel by stirring or annealing. The gel is further heated overnight at temperature of around 100 °C to form precursor. This precursor is annealed at various temperatures to get nanoparticles of different size. Wahab *et al.* utilizes this approach for synthesizing MgO nanoparticles of size ranging from 50-70 nm. These authors used magnesium nitrate as starting material and sodium hydroxide as organic host [131]. The same approach was used by Fernandez *et al.* by utilizing magnesium citrate as host [132]. Our group utilizes magnesium nitrate and host citric acid for synthesis of MgO nanoparticles in the size ranging from 9-35 nm [133]. Variation of annealing temperature provide a way to control size of nanoparticles [133,134]. We have depicted the simple process to get these nanoparticles from magnesium nitrate. SEM analysis these nanoparticles have size of almost 30 nm (see Figure 4).



Figure 6. Synthesis of MgO nanoparticles using *Artemisia abrotanum* Herba Extract. Redrawn from Iranian Journal of Science and Technology, Transactions A: Science, 42, Renata Dobrucka, Synthesis of MgO Nanoparticles Using Artemisia abrotanum Herba Extract and Their Antioxidant and Photocatalytic Properties, 547–555, Copyright (2016), from Springer Nature [144].

2.2. Ultrasonic methods

These methods utilize ultrasonic waves to make solution homogenous. The other advantage of ultrasound radiation is that, it yields smaller particles. The effects of ultrasonic radiation on chemical reactions are due to the very high temperatures and pressures that develop during the sono-chemical cavity collapse by acoustic cavitation [135, 136, 137]. Figure 5 shows a scheme that takes place during the formation of MgO nanoparticles under influence of ultrasonic irradiation [136]. MgO nanoparticles with different sizes are obtained through calcining.

The simple process of synthesizing these nanoparticles using this method is adopted by number of researchers [138, 139, 140, 141].

2.3. Green synthesis

Green synthesis techniques utilize pollutant free chemicals like as water, natural extracts for the synthesis of nanoparticles [142]. Morrthy et. al reported synthesis of MgO nanoparticles using Neem leaves [143]. Figure 6 depicts procedure for the synthesis of MgO nanoparticles using *Artemisia abrotanum* Herba Extract [144].

Some other host which are effectively utilized for synthesis of MgO nanoparticles are *Nephelium lappaceum* L. peels [145], orange fruit [146], Aqueous *Eucalyptus globulus* leaf [147] and Medicinal Plant Pisonia grandis R. Br. Leaf [148].

Other chemical methods which are widely adopted for synthesizing MgO nanostructure are facile [149,150] and micro-emulsion [151, 152, 153]. Though, chemical methods are cost effective and known to provide better control over particle size and size distribution but the particles obtained by these methods are expected to have carbecanous impurities. These kinds of impurities are reflected from near edge X-ray fine structure [154] and Fourier transform infrared spectroscopy [155, 156, 157]. Though specific methodology is adopted by researchers to remove these nanoparticles to enhance their performance [158] but still their effect cannot be neglected.

Other approaches, which are used to make nanoparticles free from carbecanous impurities, are based on solid-state reaction [159], however, synthesis of MgO nanoparticles using these methods are very limited. Kamrulzaman *et. al.* reported synthesis of MgO nanoparticles of size ranging from 20 to 135 nm from solid state reaction method by employing magnesium acetate tetrhydrate as starting material [160]. Similar approaches for synthesis of MgO nanoparticles were utilized by Zhang *et al.* (2019) [161] and Guo *et al.* (2020) [162].

3. Thin films

The other well-known form of nanostructure is thin film. Thin films are layer of material supported on other material, known as substrate. The property of thin film is determined by stoichiometric proportion, lattice mismatch [163, 164, 165, 166], nature of growth [167] and stress developed film substrate [168]. Thus, numerous methods have been developed to grow thin films of MgO by researchers, which are discussed in recent work from our group and summarized as below.

E-beam evaporation method utilizes evaporation of material target with e-beam energy. This method is used to grow MgO thin films on aircleaved (001) surfaces of LiF, NaCI, and KCI using a 6-kW electron gun [169, 170, 171]. It observed that MgO on (001) surfaces of LiF, NaCI, and KCI grows with parallel orientation in the temperature range 25–250 °C [172]. e-beam evaporation was used to grow MgO dielectric layer on glass substrate [173] as well as to grow MgO barrier layer for MgO based magnetic tunnel junctions [174] and multilayer structure [175]. This method is utilized by our group for growth of MgO thin films on quartz substrate [176] and Si substrate having thickness 5 and 50 nm [177]. The films grown on Si substrate are amorphous (5 nm) and crystalline (50 nm), in nature respectively [178]. Though films grown on quartz substrate are amorphous in nature but near edge X-ray absorption fine structure measurements revealed spectral features associated with MgO



Figure 7. O *K*-edge NEXAFS spectra of MgO thin film on quartz substrate in total fluorescence mode at 10, 45 and 70°.

formation (Figure 7). The spectral features at 532, 538, 542 and 552 eV in the O K-edge spectra at different angles, 10, 45 and 70 $^{\circ}$ C are characteristics of MgO.

Molecular beam epitaxy (MBE) provides better control over stoichiometry ratio but also helpful in epitaxial growth (Figure 7b). This technique is effectively used to grow MgO single crystals [179], epitaxial MgO films on single crystal Si(001) [180,181].

Pulsed Laser Deposition (PLD) utilizes laser beam to sputter molecules from target and used for growing epitaxial growth mode of MgO (111) films on yttrium stabilized zirconia (111) substrates [182], MgO films on Si [183] and Al₂O₃ substrates [184].

Presently most desired application of MgO is its utilization as a barrier for magnetic tunnel junction. *RF sputtering method* is preferred choice for this [185,186] as well as for other applications [80,187]. These films were prepared on a Si(001) substrate by the rf sputtering method at low ambient pressure using a metal target [186] and from MgO target [188, 189]. Our group has successfully utilized sputtering method to grow MgO thin films on quartz [190] and Si substrate [191,192]. Deposition time plays important role for controlling film thickness along with sputtering power. On the other hand, annealing improves surface of these films [193]. The nature of these films is influenced by ageing [194], annealing environment [195], irradiation [196] and implantation [197].

Deposition methods such as *chemical vapor deposition* [198, 199, 200] and *atomic layer deposition methods* [201,202] are also being utilized to grow MgO thin films. However, these methods provide better control over stoichiometry and growth, but need a dedicated instrument. Thus, cost-effective approaches for growing MgO thin films are also being developed. In this context, Yoon *et al.* utilized sol-gel method to grow MgO thin films on Si substrate [183].

4. Nanocubes

MgO nanocubes are prepared using domestic microwave oven operated at 2.45 GHz and 1000 W [203]. To synthesize these nanocubes, Mg chips and steel-wool were used as starting materials (Figure 8).

Another approach, which is utilized to synthesize MgO nanocubes is arc discharge method. Figure 9 shows schematic of arc discharge apparatus [204].

MgO nanocubes are extensively studied by group of O. Diwald [205, 206, 207, 208] by chemical vapor synthesis (CVS). This method involves a reactor consists of two concentrical quartz glass tubes placed inside a cylindrical furnace, termed as CVS reactor (Figure 10) and



Figure 8. The schematic of MgO nanocube formation. Drawn on the basis of the experimental procedure depicted in reference [203].



Figure 9. The schematic of arc discharge methods. Reprinted from Materials Letters, 65/1, Yanjie Su, Hao Wei, Zhihua Zhou, Zhi Yang, Liangmin Wei, Yafei Zhang, Rapid synthesis and characterization of magnesium oxide nanocubes via DC arc discharge, 100–103, Copyright (2011), with permission from Elsevier [204].



Figure 10. Schematic representation of the flow reactor system for the production of highly dispersed metal oxides. Redrawn from Surface Science, 290/3, E. Knözinger, Karl-Heinz Jacob, S. Singh, P. Hofmann, Hydroxyl groups as IR active surface probes on MgO crystallites, 388–402, Copyright (1993), with permission from Elsevier [209].



Figure 11. The experimental configuration RF impulse discharge plasma method. Adapted from S. Iizuka, T. Muraoka, Single-crystal MgO hollow nanospheres formed in RF impulse discharge plasmas, J. Nanomater, 2012 (2012) 691874. Copyright © 2012 Satoru Iizuka and Takumasa Muraoka [216].

utilized long-back a group of researcher long back to grow highly dispersed MgO particles [209].

Nanocubes of MgO were also synthesized by burning Mg chips and Mg ribben in air [210, 211, 212]. These nanocubes can be further transformed into nanobars under water exposure followed by vacuum annealing [213].

5. Hollow-spheres

MgO hollow nanospheres were produced via one-step laser synthesis in both gas and liquid media. In situ Kirkendall effect is responsible for the formation of the hollow nanospheres [214].

Nanoparticle-built MgO hollow microspheres were synthesized through a template-free hydrothermal route using citrate as a structural



Figure 12. Schematic illustration of the apparatus used for the synthesis of single-crystalline nanowires. Redrawn from Advanced Functional Materials, 12/4, Y. Xia, G. Zhang, Y. Yin, Synthesis and Characterization of MgO Nanowires Through a Vapor-Phase Precursor Method, 293–298, Copyright (2002), with permission from John Wiley and Sons [217].



Figure 13. TEM image of MgO nanowires with curved and branchy morphologies. Reprinted from The Journal of Physical Chemistry B, 106, C. Tang, Y. Bando, T. Sato, Oxide-assisted catalytic growth of MgO nanowires with uniform diameter distribution, 7449–7452, Copyright (2002) American Chemical Society [220].



Figure 14. High-resolution SEM image of tubular MgO nanostructures Redrawn from Inorganic Chemistry, 43, Jinhua Zhan, Yoshio Bando, Junqing Hu, et al, Bulk Synthesis of Single-Crystalline Magnesium Oxide Nanotubes, 2462–2464, Copyright (2004) American Chemical Society [221].



Figure 16. MgO nanoflowers grown using Rosmarinus officinalis L. extracts. Reprinted from Y. Abdallah, S. O. Ogunyemi, A. Abdelazez, M. Zhang, X. Hong, E. Ibrahim, A. Hossain, H. Fouad, B. Li, J. Chen, The green synthesis of MgO nano-flowers using rosmarinus officinalis L. (Rosemary) and the antibacterial activities against Xanthomonas oryzae pv. oryzae, BioMed Res. Inter. 2019 (2019) 5620989. Copyright © 2019 Yasmine Abdallah et al [231].

director. Zn was introduced into MgO to improve the surface charge [215]. Lizuka and Muroka use RF impulse discharge plasma for synthesis of MgO hollow sphere [216]. Figure 11 shows the schematic of equipment used for synthesizing hollow sphere using this method.

6. Nanowires

Nanowires are most common form other than nanoparticles and thin films, which get more attention by researchers. Yin *et al.* reported a vapor phase method for generation of MgO nanowires from MgB₂ source. Figure 12 shows the schematic of formation of MgO nanowires by this group [217].

The growth of MgO nanowires from the vapor liquid-solid (VLS) growth mechanism with Mg_3N_2 as a precursor [218] as well as from MgO/Si with Au catalysts [219]. Figure 13 shows the MgO nanowire grown using boron oxide-assisted catalytic method from metal magnesium [220].

7. Nanotubes

Crystalline tubular magnesium oxide nanostructures were obtained through carbon-thermal evaporation of a MgO powder while adding Gallium oxide into the mixture of MgO and carbon (Figure 14) [221]. Znassisted catalysts-free method is utilize to grow MgO nanotubes by thermal evaporation of mixed Zn and Mg powders [222]. These nanotubes are also grown by other researchers with various methods of depositions [223, 224, 225].



Figure 15. Schematic illustration of the synthesis of MgO nanorod. Redrawn from Materials Research Bulletin, 35/10, Z Cui, G.W Meng, W. D Huang, G. Z Wang, L.D Zhang, Preparation and characterization of MgO nanorods, 1653–1659, Copyright (1993), with permission from Elsevier [228].



Figure 17. (a) SEM and (b) TEM images of MgO Nanobelts. Reprinted from Materials Letters, 102, Hongji Li, Mingji Li, Xiufeng Wang, Xiaoguo Wu, Fude Liu, Baohe Yang, Synthesis and optical properties of single-crystal MgO nanobelts, 80–82, Copyright (2013), with permission from Elsevier [235].

8. Nanorods

MgO nanorods with spherical particles at the tips and having diameters ranging from 20 to 50 nm were fabricated through direct reaction of Mg and oxygen. The lengths of these nanorods are observed up to 10 μ m [226]. The controlled growth of MgO nanorods was investigated under electron irradiation in TEM [227]. Synthesis of magnesium oxide (MgO) nanorods was achieved by simply heating MgCl₂ powder at 750 °C for 1.5 h in a constant flow of mixture gas (90% pure argon and 10% pure hydrogen) as shown in Figure 15 given below [228]. Vapor-liquid-solid (VLS) mechanism is also MgO nanorods have been grown on Si (100) by using thermal evaporation of Mg₃N₂ powders [229].

9. Nanoflower

Nanoflowers are among the most studied nanostructure of MgO and receive significant interest for understanding of growth phenomena [230]. In a recent study, these nanoflowers are grown using *Rosmarinus officinalis L*. extracts (Figure 16) [231] as well as from magnesium chloride with the help of acacia gum [232].

Zheng *et al.* grown MgO nanoflowers by taking magnesium nitrate and sodium carbonate as starting material [233]. Nano-MgO films prepared using sol-gel spin coating method converted to flower-like structure after annealing [234].

10. Some other special structure

Li *et al.* reported the growth of MgO nanobelts by the decomposition of magnesium nitrate in direct current arc plasma jet chemical vapor deposition process with Mo substrate at 950 °C. The process results in the formation of nanobelts after the process 0.5 min (Figure 17) [235].

First observation of MgO nanowall structures were reported on the glass substrate. These nanostructures are grown using successive ionic layer adsorption and reaction (SILAR) method followed by annealing [236]. MgO based nanoplates [237, 238, 239], nanosheet [240,241], nanofibers [242,243] are other form of nanostructures of significant interest.

11. Synthesis of nanoparticles using advanced techniques

Thus, variety of methods have been discussed for synthesis of MgO nanostructures, however, nanostructures grown using chemical methods usually contain carbonaceous impurities. These nanostructures when grown using physical methods contain surface defects as well as stress. These several effects lead to tailored behavior of these nanoparticles. Hence, attempts are also made by researchers to utilize self-sustained growth of nanoparticles under influence of sufficient energy. The energy to initiate grain growth is provided either by X-ray source [244], or heavy ion irradiation [245].

These methods are able to make these nanostructures free from carbonaceous impurities as well as strain. Thus, monochromatic X-ray by Bharti *et al.* [246] reported synthesis of plasmonic nanostructures. However, reports depicting the formation of oxides using these methods are hardly reported. Thus, these tools open pathways to synthesize oxide nanoparticles.

12. Conclusion(s)

In this review, description of underlying phenomena observed in MgO nanostructures are briefly discussed. A description of their applications in different field of these nanostructures is described. It is contemplated that these nanostructures have different categories like nanoparticles, thin films, nanowall, nanobelt etc depending upon method of synthesis. Thus, a precise control over method of synthesis is highly desirable. Though, chemical methods are cost effective and able to design nanostructure of desired category but they lead to presence of carbonaceous impurities. Nanostructures in the form of thin films are grown using deposition methods like pulsed laser deposition, radio frequency sputtering etc. However, these methods of depositions induces strain in the films. From the future development in this research area, some advanced techniques based on synchrotron radiation and swift heavy ion irradiation may be the effective synthetization tools for these nanostructures.

Declarations

Author contribution statement

All authors listed have significantly contributed to the development and the writing of this article.

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Competing interest statement

The authors declare no conflict of interest.

Additional information

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