



# Synthesis of mixed calcite–calcium oxide nanojasmine flowers

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## Abstract

Here we report for the very first time a simple, inexpensive, room temperature synthesis method of formation of mixed calcite–calcium oxide nanojasmines (CTCaONJs). The method involves chemical precipitation in aqueous medium at room temperature in open atmosphere in order to allow natural carbonation to occur. Aqueous solutions of calcium nitrate dihydrate ( $\text{Ca}(\text{NO}_3)_2 \cdot 2\text{H}_2\text{O}$ ) and sodium hydroxide are used as precursors. The nanopowder is characterized by the Fourier transform infrared (FTIR) spectrometry, X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM) and transmission electron microscopy (TEM), energy dispersive X-ray analysis (EDAX), BET (Brunauer–Emmett–Teller), BJH (Barrett–Joyner–Halenda) techniques. Optical properties of nanojasmines (NJs) were investigated by UV–vis spectroscopy. The powder has calcite as the major phase and calcium oxide as the minor phase. The crystallite size of calcite along (104) crystallographic direction is about 18 nm with a size strain of about 1.03%. The high (e.g.,  $8.8 \text{ m}^2 \text{ g}^{-1}$ ) surface area powder exhibits a mesoporous microstructure with 5–15 nm pore size. Further, it possesses a relatively higher optical band gap of 5.87 eV. The FESEM and TEM based evidences show that the CTCaONJ flowers mainly comprise of nanoplatelets made up of irregular nanorods and nanowires consisting of amorphous spherical nanoparticles. Based on these experimental evidences a qualitative model is proposed to explain the growth mechanism of the CTCaONJ flowers.

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**Keywords:** Calcite–calcium oxide; Nanojasmine; Flowers; Aqueous medium; Growth mechanism

## 1. Introduction

One dimensional (1D) nanomaterials are extremely important for advanced optical and semiconducting devices, sensors, nanoelectromechanical devices, catalyst, supercapacitors, bio-imaging probes, microwave absorbers, energy devices and toxic waste management [1–4]. Self-assembled nanostructure of such 1D nanomaterials results in the evolution of petals or flower like morphology [5–10]. Among the various morphologies the 3D nanoflowers attract more attentions because of

their robust structure, huge internal space, and large specific surface area [11–13]. Such unique nanostructure with lots of pores on petals displays a great potential in applications such as gas sensing, toxic waste remediation, scavenging of divalent and tetravalent heavy metals, removal of low-concentration  $\text{NO}_x$ , flame retardation, catalysis, energy storage and so on [14–21]. Numerous research efforts are hence devoted to the controllable preparation of such nanostructures with diverse morphologies and hierarchical structure [14–33].

A typical representative survey of pertinent literatures [22–33] on synthesis of nanoflowers of different materials e.g.,  $\text{MgO}$ ,  $\text{SnO}_2$  etc. is summarized in Table 1. The results presented in Table 1 point to a few generic features. The first generic feature is that the synthesis procedures used are mostly very difficult to perform, and usually require high temperature and pressure e.g., conventional evaporation [22,27], chemical vapor deposition [23],

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Table 1  
Literature survey on synthesis of nanoflowers.

Synthesis route	Inorganic precursors	Synthesis medium	P/T (°C)	Brief processing conditions	Phase (s) <sup>a</sup>	PS (nm)	Remarks	Reference
CE	Mg powders and distilled water	Aqueous medium	950	Used deposition substrate: alumina tube protecting medium: Ar gas	FCC MgO	~Diameter 20–80 nm	Star like NFs of MgO consist of Nfbs	[22]
CVD	Zn powder and SiC plate with Si face upturned	Vapor phase	480–600	Protecting medium: 150 sccm Ar gas mixed with 3–5 sccm of O <sub>2</sub>	Hexagonal wurtzite ZnO	~1–2 μm	Rose like ZnO NFs composed of many curled and wrapped layered NS	[23]
HT	BHMTA and Zn (NO <sub>3</sub> ) <sub>2</sub> · 6H <sub>2</sub> O, TiO <sub>2</sub> NPs	Aqueous medium	140 °C for 2 h	Slurry was placed in a Teflon like autoclave, physical blending of TiO <sub>2</sub> NPs	Pure pristine ZnO, rutile phase of TiO <sub>2</sub>	~Micron size	Composite TiO <sub>2</sub> /ZnO NFs has better photocatalytic activity	[24]
HT	BHMTA, Zn(NO <sub>3</sub> ) <sub>2</sub> · 6H <sub>2</sub> O, TiO <sub>2</sub> NPs and AgNO <sub>3</sub> soln.	Aqueous/organic medium	140 °C for 2 h	Slurry was placed in a Teflon like autoclave, physical blending of AgNO <sub>3</sub> solution, TiO <sub>2</sub> NPs	Pure pristine ZnO, rutile phase of TiO <sub>2</sub> , metallic Ag	~Micron size	Composite Ag/TiO <sub>2</sub> /ZnO NFs with much better photocatalytic activity	[25]
CP	MgCl <sub>2</sub> · 6H <sub>2</sub> O, NaOH and AG	Aqueous medium	500 °C	10%, 20% solution of AG are used for MgO modification	FCC MgO	~40–70 nm	Highly efficient MgO NFs for removal of divalent metallic species from waste water	[26]
CE	Mg powder, Si wafer	Dry evaporation	651–850 °C	Control heating of Mg powder placed on surface of Si wafer	–	Length ~micron, diameter ~20–80 nm	MgO NFs, novel hierarchical MgO NSt with [fourfold symmetry]	[27]
TP	DBTDL and Ti plates	Organic/acidic aqueous medium	350 °C for 1.5 h	Flow rate: ultrapure Ar at 100 sccm on etched Ti plate	Cassiterite SnO <sub>2</sub>	NPEs ~50 n, Holes diameter ~10–100 nm	3D Sn NFs were synthesized on favorable Ti Surface	[28]
TC/PR	SnS or SnS <sub>2</sub> , SnCl <sub>4</sub>	Organic medium	350–550 °C	Placing the SnS or SnS <sub>2</sub> NFs precursors, grown via a polyol refluxing process	Pure tetragonal SnO <sub>2</sub>	~2 μm	porous SnO <sub>2</sub> and Pt/SnO <sub>2</sub> NFs with enhanced gas sensing performances	[29]
SR	In(AC) <sub>3</sub> and en	Aqueous/organic medium	240 °C for 18 h	Precursor soln. are placed in a Teflon like high pressure autoclave for 18 h	BCC In(OH) <sub>3</sub>	~90 nm	NShs, NFs of In(OH) <sub>3</sub> , In <sub>2</sub> O <sub>3</sub>	[30]
HT	NaOH, Zn(NO <sub>3</sub> ) <sub>2</sub> · 6H <sub>2</sub> O, and TiO <sub>2</sub> powders	Aqueous medium	120 °C for 10 h	Precursor solution are placed in a Teflon like high pressure autoclave for 10 h	Orthorhombic H <sub>2</sub> Ti <sub>2</sub> O <sub>5</sub> · H <sub>2</sub> O	–	Numerous flower like aggregates of titanate are formed	[31]
IG/BM	CS, CaCl <sub>2</sub> · 2H <sub>2</sub> O, NaTPP, CH <sub>3</sub> COOH, H <sub>2</sub> O <sub>2</sub> , THMAM and FITC	–	–	Overnight incubation followed by dialysis against buffer for 72 h	Calcium pyrophosphate hydrate phase	~Micron size	–	[32]
SSC/CP	CaCO <sub>3</sub> , Cu <sub>2</sub> CO <sub>3</sub> · 2H <sub>2</sub> O and CoCO <sub>3</sub>	Solid state/ aqueous medium	900 °C for 2 h	Both in aqueous soln and solid state precursor are mixed and calcined at particular temp.	Dicalcium dicobalt oxide phase, tri calcium dicobalt oxide phase	~50 nm to 1 μm	Mixed metal oxides NFs, with enhances the charge storage ability	[33]

<sup>a</sup>Qualitative assessment of phases, T: Process temperature, CE: Conventional evaporation, CVD: Chemical vapor deposition, HT: Hydrothermal treatment, CP: Chemical precipitation, TP: Thermal pyrolysis, TC/PR: Thermal calcination/polyol refluxing, SR: Solvothermal route, IG/BM: Ionotropic gelation with biomimetic mineralization, SSC/CP: Solid-state calcination and co-precipitation routes, Mg: Magnesium, Zn: Zinc, BHMTA: bis-hexamethylene triamine, Zn(NO<sub>3</sub>)<sub>2</sub> · 6H<sub>2</sub>O: Zinc nitrate hexahydrate, TiO<sub>2</sub> NPs: Titania nanoparticles, AgNO<sub>3</sub>: Silver nitrate, AG: Acacia gum, MgCl<sub>2</sub> · 6H<sub>2</sub>O: Magnesium chloride hexahydrate, NaOH: Sodium hydroxide, Si: Silicon DBTDL: di-butyl tin di-laurate, SnS: Tin sulfide, SnS<sub>2</sub>: Tin disulfide, SnCl<sub>4</sub>: Stannic chloride, In(AC)<sub>3</sub>: Indium acetate, en: ethylene diamine, CS: Chitosan, CaCl<sub>2</sub> · 2H<sub>2</sub>O: Calcium chloride hydrate, NaTPP: Sodium tripolyphosphate, CH<sub>3</sub>COOH: Glacial acetic acid, H<sub>2</sub>O<sub>2</sub>: Hydrogen peroxide, THMAM: tris (hydroxyl methyl) amino-methane, FITC: Fluorescein isothiocyanate, CaCO<sub>3</sub>: Calcium carbonate, Cu<sub>2</sub>CO<sub>3</sub> · 2H<sub>2</sub>O: Copper carbonate hydrate, CoCO<sub>3</sub>: Cobalt carbonate, NFs: Nanoflowers, Nfbs: Nanofibres, NS: Nanosheets, NSt: Nanostructures, NPEs: Nanopetals, NShs: Nanospheres.

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