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Size Stabilizers in Two-electrode Synthesis of ZnO Nanorods

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We modify and optimize a cheap, simple and effective synthesis of zinc oxide nanosized particles by electrodeposition. The core method encompasses the synthesis of ZnO product on the soluble zinc anode of the two-zinc-electrode cell emerged in aqueous NaCl. Resulting particles have the plate shape, wider in the middle and sharp at the edges. They have uniform shape, but broad size distributions with most of the ZnO product 1 - 2 μm long and 0.5 - 0.7 μm wide. Thus, auxiliary stabilizers are added to aqueous phase to reduce the size and narrow its distribution in the target product. Here we present the size stabilizing action of four successful stabilizers: urea, polyvinyl alcohol, Triton X-100 and Atlas G3300. All of them reduce particle size and polydispersity. An anionactive surfactant Atlas G3300 is the most effective, giving an order of magnitude nanorod size reduction.

Keywords: ZnO, nanorods, electrodeposition, oxidation, surfactants.

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Introduction

1D semiconductor nanostructures (nanorods and nanowires) already play an important role as both interconnects and functional units in the fabrication of electronic, optoelectronic, electrochemical, and electromechanical nanodevices [1]. It is also argued that their unique properties make them great building blocks for the new generations of electronic and photonic devices [2].

ZnO is a semiconductor material with a direct wide band gap energy (3.37 eV) and a large exciton binding energy (60 meV) at room temperature [3]. It crystallizes in two main forms, hexagonal wurtzite, and cubic zinc blende. A comprehensive review of properties, preparation, processing, and device applications of ZnO is presented elsewhere [4]. Here we only briefly motivate the development of synthetic methods aiming at ZnO wurtzite nanorods by noting their significance.

ZnO nanoparticles are attractive candidates for various technological applications such as UV lasers [5],

light-emitting diodes [6], solar cells [7] and stills [8], nanogenerators [9], gas sensors [10,11], UV photodetectors [12], and photocatalysts [13-16]. ZnO is also biocompatible and biodegradable [17], and therefore suitable for medical [18] and environmental applications [19]. Most interestingly, the properties of zinc oxide as a material strongly depend on the dimensionality, size and morphology of the ZnO nanostructures [13,20-22].

Nowadays there is a multitude of synthetic methods to obtain ZnO. To name a few, vapor-phase transport [23], sol electrophoretic deposition [24], hydrothermal/solvothermal growth [3,16,25], arc discharge [26], chemical bath deposition [12,27,28], or electrochemical deposition [29-31] (both anodic [32-34] and cathodic [35,36]). They differ in their approach, cost, and ultimate results. The two-electrode neutral aqueous synthesis is simple, green and cheap, gives high yields and allows the current-time control of product quantity [20,30,32,34,37-39]. It is successfully used for the manufacture of ZnO thin films [40]. It is also suitable for electrochemical synthesis of 1D ZnO

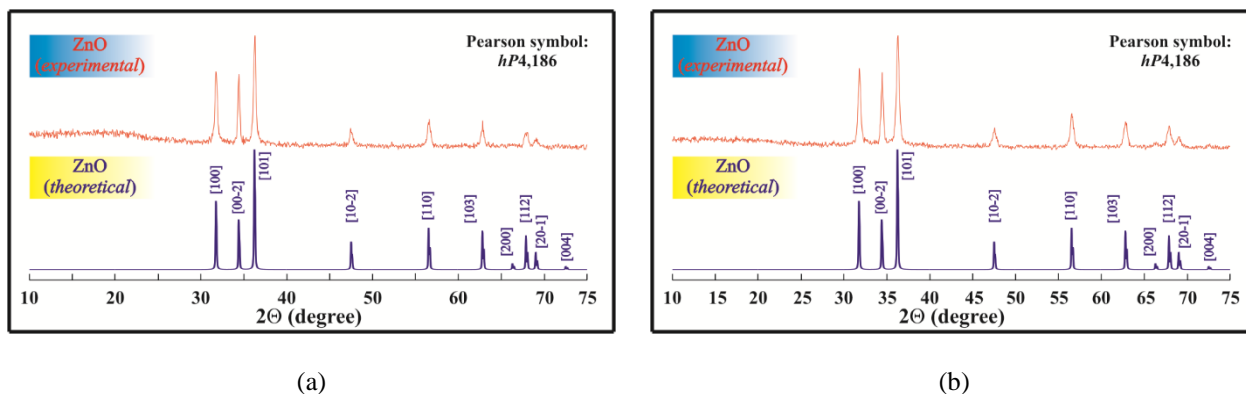


Fig. 1. XRD spectra of the ZnO products (experimental in red) (a) without stabilizers and (b) synthesized with Atlas G3300. These are super imposed on the theoretical $P6_3mc$ wurtzite ZnO peaks (shown in blue).

Table 1

Conditions of Synthesis and corresponding yields

Current density mA/cm ²	25	50	75	100	125
control	1A (93%)	1B (91%)	1C (92%)	1D (92%), 1D* (91%), 1D+ (90%)	1E (93%)
urea	2A (94%)	2B (91%)	2C (90%)	2D (95%), 2D+ (90%)	2E (93%)
PVA	3A (94%)	3B (93%)	3C (95%)	3D (94%), 3D+ (93%)	3E (95%)
Triton X-100	4A (93%)	4B (91%)	4C (90%)	4D (94%)	4E (93%)
Atlas G3300	5A (93%)	5B (94%)	5C (91%)	5D (93%), 5D* (92%), 5D+ (94%)	5E (94%)

Unmarked syntheses are 5 minutes long, * signifies 10-minute and + marks a 15-minute synthesis.

nanostructures [20,32,37,38]. Current density [29] and electrolysis time [41] have been shown to impact ZnO microstructures in other electrodeposition methods. Earlier we also [38] examined how electrolysis time and current density affects the product's topology in our set-up and as a result changes its photo-induced piezoelectric properties.

This article reports further method development: we are testing four candidates for the size stabilizing precursors in a range of synthetic conditions. Hydrothermal syntheses of ZnO with the use of solvents [8], polymers [42-44], salicylic acid [45], amino acids [46] and surfactants [3] were reported earlier. Our current choice of stabilizers tested is not random. The compounds that we are testing were used earlier in different synthetic procedures aiming at ZnO: urea [47] as an alkali source together with N_2H_4 and as a stabilizer [48], PVA [49-51]. Surfactants that are tested represent two different groups: non-ionic Triton X-100 and anionic Atlas G3300 (formulas and chemical names can be found in Figure 5). To the best of our knowledge only polymetacrylic acid and cationic surfactant CTAB [52] were tested as stabilizers in an analogous synthetic procedure earlier.

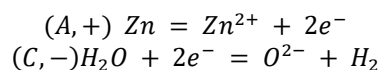
ZnO synthesis by anodization is studied only fragmentarily and the mechanism is not completely understood [32]. There are many other factors affecting the ZnO product: temperature [53], electrolyte type and concentration [30,32,53] and even the orientation of electrodes [32]. Optimization of synthetic conditions is therefore promising, but extremely challenging with a multitude of factors to control.

I. Main Text

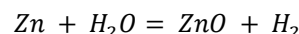
In the electrolytic cell



the following half-reactions occur on the electrodes:



The total equation for electrolysis then reads:



1.1. Characterization of the ZnO product

Dried particles are analyzed using XRD and the experimental peaks are in perfect agreement with $P6_3mc$ wurtzite phase ZnO (Fig. 1). These results are consistent with earlier reports of zinc oxide synthesis via electrodeposition at high temperatures in aqueous neutral electrolyte [20,37,38]. The XRD spectra of unstabilized particles (panel a) and those made in the presence of each of the four stabilizers (product of the synthesis with Atlas G3300 is given on panel b as an example) are essentially identical (XRD spectra of with other). Therefore, the presence of stabilizers affects neither chemical composition nor crystalline phase of synthesized particles. Nonetheless, peaks on panel (b) are significantly broader compared to panel (a), indicating the smaller particle size of the latter.

Percentage yields are calculated based on the decrease of the mass of the anode and presented in the Table 1. These are pleasantly but not unusually high

Table 2

Average geometric sizes of the particles synthesized at different current densities with the stabilizers tested

Conditions	A	B	C	D	E
1, control	944 ± 264 ^a	1459 ± 438	1449 ± 503	1804 ± 769	1713 ± 719
	320 ± 64 ^b	448 ± 103	443 ± 169	518 ± 164	501 ± 155
2, urea	862 ± 288	691 ± 309	828 ± 392	560 ± 270	942 ± 270
	230 ± 68	201 ± 83	260 ± 101	214 ± 66	259 ± 76
3, PVA	496 ± 196	1162 ± 457	860 ± 240	870 ± 351	927 ± 384
	210 ± 81	337 ± 99	360 ± 113	298 ± 107	315 ± 96
4, triton x-100	729 ± 292	1082 ± 335	1170 ± 461	1129 ± 393	1126 ± 307
	259 ± 63	312 ± 84	336 ± 111	357 ± 148	344 ± 75
5, atlas	238 ± 125	177 ± 40	119 ± 45	109 ± 45	188 ± 46
	79 ± 50	51 ± 28	35 ± 10	34 ± 9	62 ± 32

^a mean particle lengths; ^b mean width measured at the center of each particle. Both are reported with the standard deviation, in the form $\mu \pm \sigma$ nm.

Table 3

Average geometric sizes of the particles synthesized at different electrolysis times with the stabilizers tested

Conditions	D	D*	D+
1, control	1804 ± 769	1068 ± 468	900 ± 392
	518 ± 164	369 ± 119	266 ± 107
2, urea	559 ± 270	-	892 ± 355
	214 ± 66	-	287 ± 132
3, PVA	870 ± 351	-	700 ± 305
	298 ± 107	-	122 ± 110
5, atlas	109 ± 45	110 ± 46	104 ± 50
	34 ± 9	34 ± 9	35 ± 16

(compared to electrodeposition of other metal oxides and salts).

There are 5 series of experiments conducted, one with no stabilizer added (control) and four more (with each of 4 stabilizers). The total of 31 sets of conditions were tested to examine the influence of the stabilizers, current density (Table 2) and electrolysis time (Table 3) on the size and morphology of the ZnO product.

The unstabilized ZnO particles (syntheses 1A to 1E) have the plate shape, wider in the middle and sharp at the edges (figure 2). The size distribution is wide (row 1 of tables 2 and 3) with most of the synthesized particles being more than $1\mu\text{m}$ in length, but there is also a significant number of smaller particles (see figure 2).

The average particle size increases with higher current density (row 1 of table 2). This increase in average particle size is due to the reduced fraction of smaller particles at higher current densities (compare panels (a)-(c) of Figure 2). The positive correlation between current density and particle size can be explained by the faster product formation, leading to more aggregation due to a higher density of ZnO clusters.

Increasing the electrolysis time leads to smaller particles in the unstabilized syntheses (table 3, row 1 and panels (c),(d) in Figure 2).

1.2. Effect of stabilizers

The first thing to note in Tables 2 and 3 is that all four of the stabilizers tested result in a decrease of the average particle sizes for all the conditions tested. One might expect that the stabilizers will be less effective when the rate of the synthesis is higher (at higher current densities). However, they remain effective over the full range of current densities tested. PVA and Triton X-100 predictably give smallest particles at lowest current density (mirroring the trend observed in the case without the stabilizer). Urea and Atlas G3300, on the other hand, seem to have an optimal current density of around 100 mA/cm^2 .

In the control syntheses increasing electrolysis time led to the decrease of the average particle size (table 3 and Figure 2). So another important question one might ask is whether the stabilizers are as effective for longer syntheses. If the stabilization is kinetic, it can be undermined over time by aggregation, and/or reaching the equilibrium size.

This is indeed what is happening with urea. When the electrolysis is 5 minutes long, there is a considerable size reduction (see Table 3 and compare Figures 2(c) and 5(a)). However, the 15-minute syntheses are unaffected by the presence of urea (column D+ of table 3, compare Figures 2(d) and 5(b)).

On the other hand, PVA decreases the average size

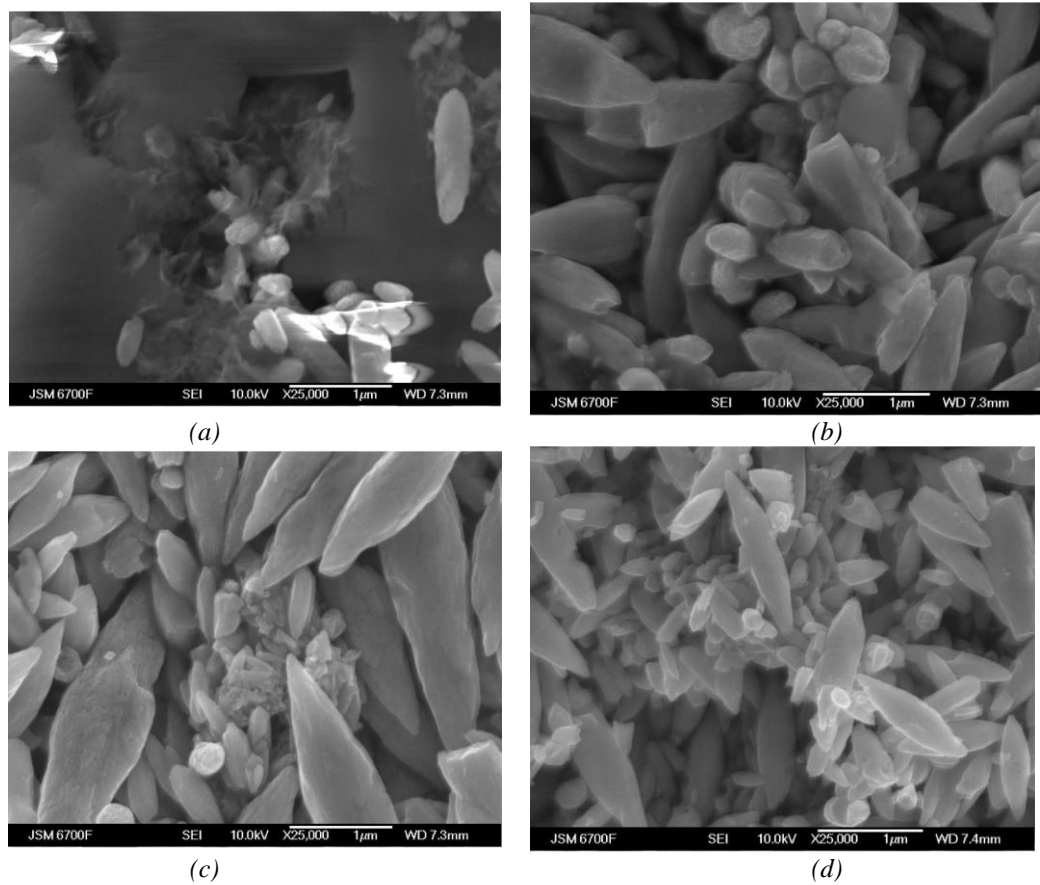


Fig. 2. ZnO nanoparticles synthesized without stabilizers at the following conditions: (a) 1A (25 mA/cm^2 , 5 min), (b) 1B (50 mA/cm^2 , 5 min), (c) 1D (100 mA/cm^2 , 5 min), (d) 1D+ (100 mA/cm^2 , 15 min).

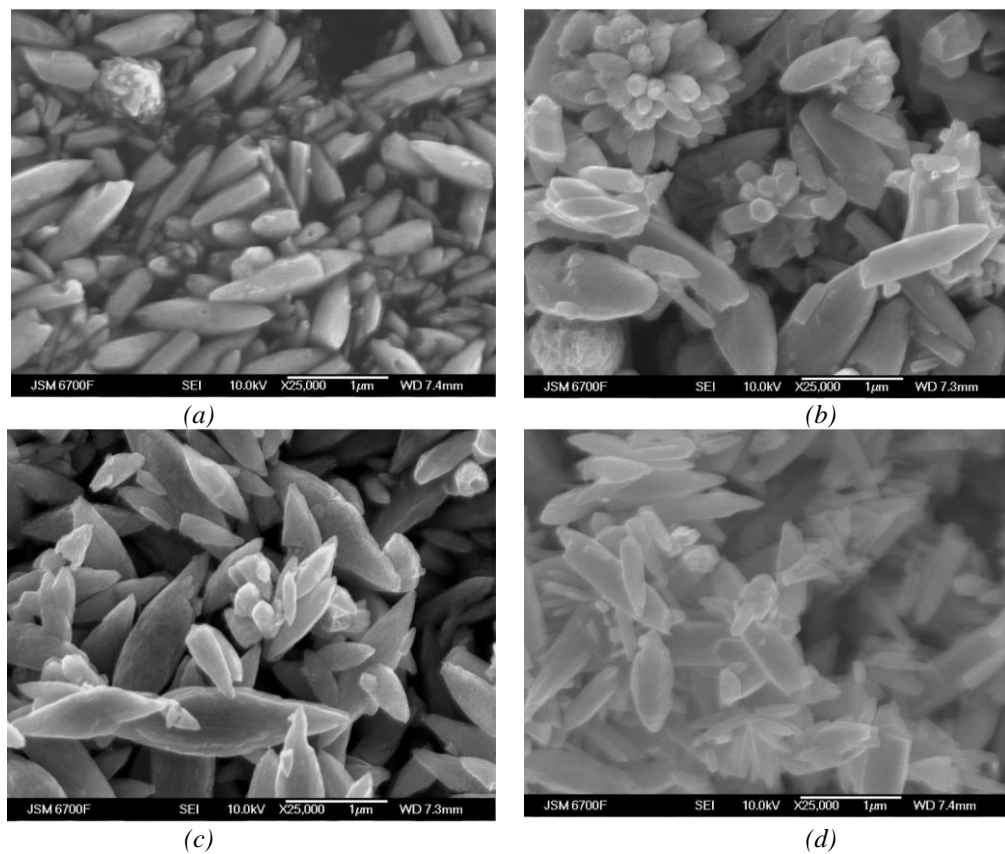


Fig. 3. Stabilized ZnO nanoparticles: (a) 2D, (urea, 32 mA/cm^2 , 5 min), (b) 2D+ (urea, 32 mA/cm^2 , 15 min), (c) 3D (PVA, 32 mA/cm^2 , 5 min), (d) 3D+ (PVA, 32 mA/cm^2 , 15 min).

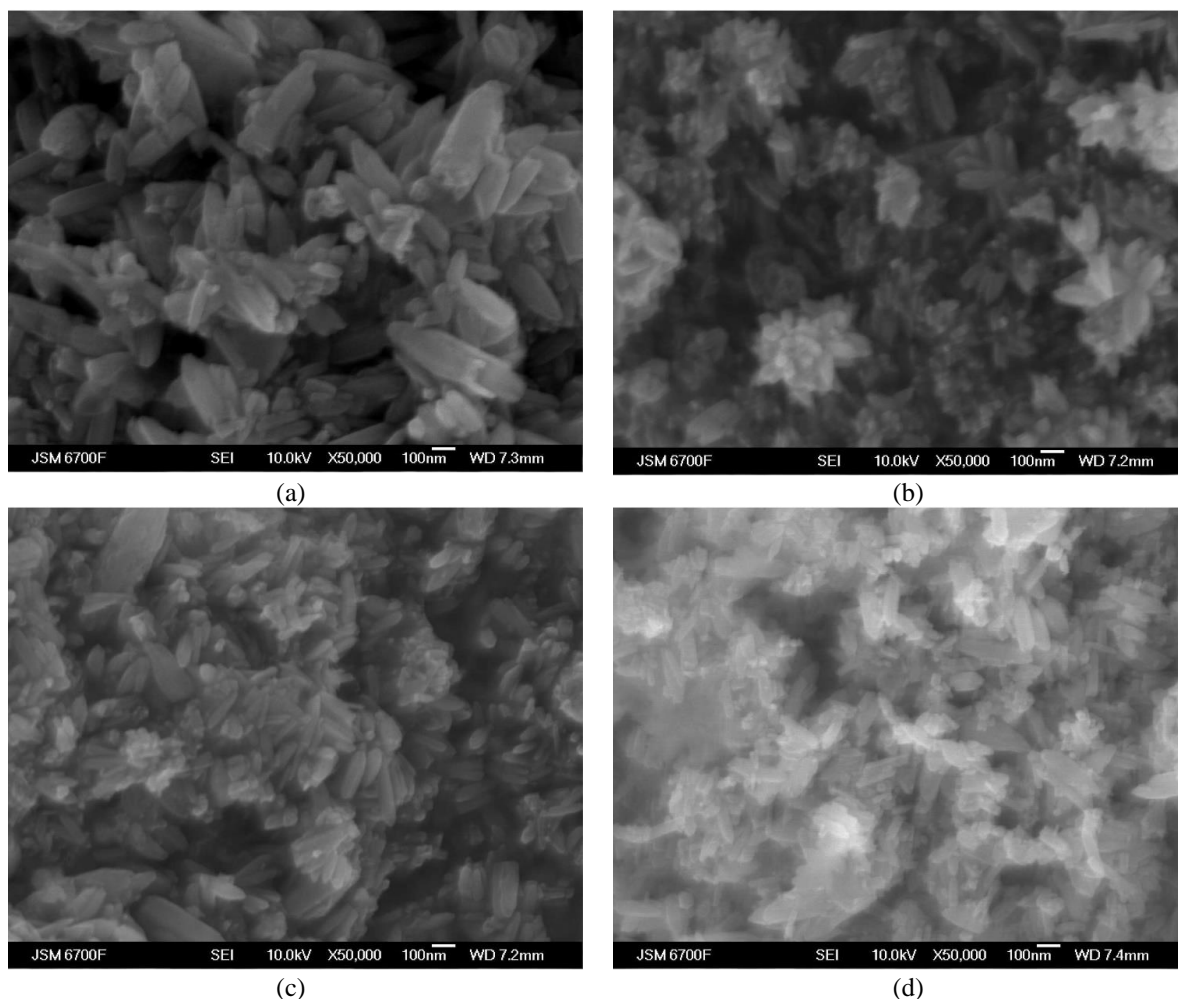


Fig. 4. ZnO nanoparticles stabilized with atlas: (a) 4A, (25 mA/cm^2 , 5 min), (b) 4C (75 mA/cm^2 , 5 min), (c) 4D* (100 mA/cm^2 , 10 min), (d) 4D+ (100 mA/cm^2 , 15 min).

in both short (compare Figures 2(c) and 5(c)) and long (Figures 2(d) and 5(d)) syntheses.

The findings with Atlas G3300 are the most exciting (Fig. 4). It gives consistently small particles independent of the length of the synthesis. In addition to be suitable for both short and long syntheses, it reduces the size of ZnO particles by about 10 times (compared to the unstabilized syntheses) in all the conditions tested (note that the magnification is 25000 on figures 2 and 5 and 50000 on Figure 4). The use of Atlas G3300 as a stabilizer results in a product with a narrow size distribution. In addition to decreasing the particle size, Atlas G3300 also changes the shape of the product. The particles are now more rod-like, less pointy at the edges.

The effect on particle size and morphology is repeatable as demonstrated by replicating the results of this study.

Earlier we found that flower-like ZnO aggregates have much higher piezoelectric sensitivity coefficients [38]. There are other studies reporting superior properties of flower-like ZnO nano-aggregates [11,14-16]. It is therefore worth noting here, that urea and Atlas G3300 facilitated the formation of flower-like aggregates at small current densities (Figures 5(b) and 4(b)). Unfortunately, the optimal conditions to promote the formation of the flower-like structures are not yet clear. The aggregates are,

however, sensitive to both current density and electrolysis time.

II. Experimental

Materials: 99.9 % rectangular zinc purified by galvanization was used and distilled water was used all throughout. NaCl, Atlas G3300 and urea were obtained from Sigma-Aldrich, 97 % Triton X-100 ($n = 6 - 8$) was purchased from MERX, and chemically purified grade PVA ($n \approx 200$, MW about 15 kDa).

The structural formulas of the stabilizers used are given below:

ZnO nanorod synthesis: ZnO nanorods were synthesized in a two-electrode 50 g/L (0.856 mol/L) NaCl-aqueous system with pure (99.9 %) rectangular zinc electrodes. The temperature was maintained at $363 \pm 2\text{K}$. B5-46 AC/DC converter was used to generate direct current. Solutions (except for control) contained one of the four stabilizers tested with the following concentrations: 10 g/L urea, 2 g/L PVA, 120 mg/L (saturated) Triton X-100, and 1 g/L Atlas G3300. Each stabilizer was tested at five current densities (25, 50, 75, 100 and 125 mA/cm^2) and up to three electrolysis times (5, 10 and 15 minute) giving a total of 31 sets of conditions (see Table 1).

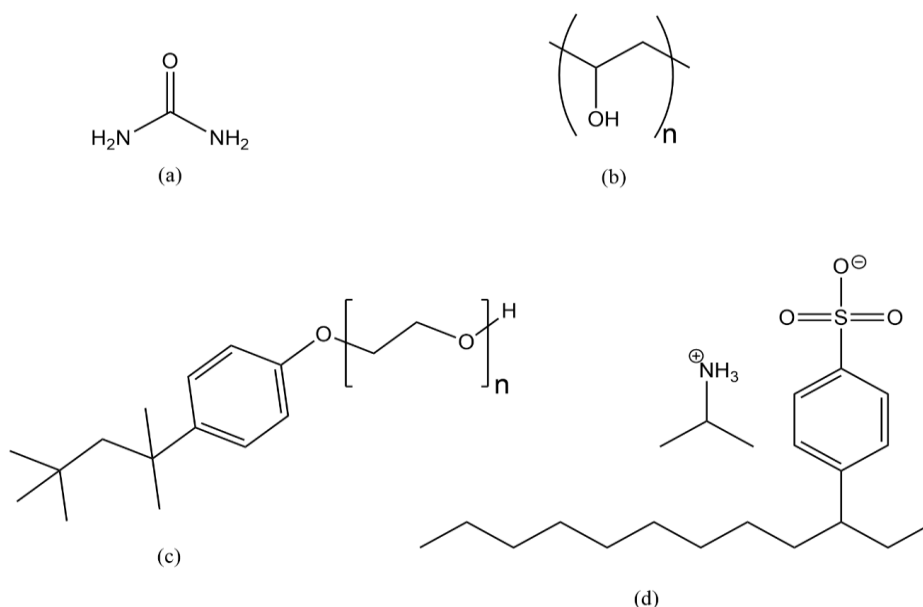


Fig. 5. Stabilizers, tested in this study. (a) urea, (b) polyvinyl alcohol, (c) Triton X-100 or 2-(4-(2,4,4-trimethylpentan-2-yl)phenoxy)oligoepoxy-ethan-1-ol, (d) Atlas G3300 or 4-dodecan-3-ylbenzenesulfonic acid;propan-2-amine.

Synthesized ZnO was decanted, washed with distilled water and dried in the oven at 50 °C overnight. Some experiments were repeated to test for the reproducibility. The differences in particle size distributions of replicate samples were not statistically significant.

Particle characterization: Powder X-ray diffraction was carried out using DRON 4-13($Cu K_{\alpha}$, Si as ethalon, 41 kV, 21 mA) on the range 2θ of 10 to 75°. Obtained spectra were compared to ZnO, $Zn(OH)_2$, Zn in CSD.

SEM of ZnO was performed using the field emission scanning electron microscope (SEM) JSM-6700F produced by JEOL Ltd. (Japan). Morphology of the powders was investigated in secondary electron imaging (SEI) mode at 100-200 pA beam current and 10 kV accelerating voltage. The vacuum level inside the SEM specimen chamber was at least 9.3×10^{-5} Pa.

Conclusion

Addition of size stabilizers improves the synthetic prospective of a 2-electrode electrodeposition of ZnO significantly. In addition to the simplicity, high yields and low cost, inherent in the method, it also acquires a way to control the shape and size of ZnO particles synthesized

electrochemically. In particular, Atlas G3300 should be the stabilizer of choice to make uniformly sized 100 to 200-nm long ZnO nanorods (depending on current density).

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Двохелектродний синтез наночастинок ZnO в присутності різних стабілізаторів

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Модифіковано та оптимізовано дешевий, простий та ефективний синтез нанорозмірних частинок цинку оксиду шляхом електроосадження. Метод полягає у синтезі ZnO в електродній чарунці з двома цинковими електродами та водним розчином натрій хлориду. Утворені частинки мають пластинчасту форму: розширені посередині і звужені по краях. Частинки мають однакову форму, але широкий розподіл за розміром з більшістю частинок ZnO довжиною 1 - 2 мкм і товщиною 0,5 - 0,7 мкм. Стабілізатори додають у водну фазу, щоб зменшити розмір і звужити його розподіл у цільовому продукті. В цій роботі ми представляємо стабілізуючу дію чотирьох таких стабілізаторів: сечовини, полівінілового спирту, Тритону X-100 та атласу. Усі вони зменшують розмір частинок і полідисперсність. Аніонний поверхнево-активний Атлас G3300 є найефективнішим, і забезпечує зменшення розміру наночастинок на порядок.

Ключові слова: ZnO, наночастинок, електроосадження, окислення, ПАВ.