SbVO₄ nanoparticles synthesized via three facile one-pot methods: controllable morphologies and superhydrophobic coatings

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The inert colloidal SbVO₄ with special morphologies is promising in practical superhydrophobic coatings. However, its conventional synthesis suffers from harsh preparation conditions, yet limiting the material applications. Herein, by using K₆V₁₀O₂₈·9H₂O as a novel vanadium source, the SbVO₄ nanoparticles were prepared through three different facile methods of hydrothermal method, reflux method and ultrasonic method. The pH values of reacting environment were tuned as the only variable to shape the final products. Colloidal morphologies can be engineered to microflakes, or amalgamations of nanoparticles. Drop-casting glasses with the as-synthesized samples will lead the surface superhydrophobicity under basic, neutral, and acidic conditions. Static contact angle of as-coated film can reach above 160° and durable even under salt solution of different pH values. Besides creating anticorrosive and hydrophobic coating materials, these facile synthesis routes may also facilitate large-scale manufacture of similar chemicals for practical applications.

Introduction

Physicochemical properties of inorganic colloidal nanocrystals exhibit high size- and shape-dependences within a scale of 1 – 10 nm due to quantum confinement effects [1–4]. A typical example stays within the family of antimony vanadium oxides, including variations of Sb_{0.92}V_{0.92}O₄ [5], Sb₂V₂O₉ [6], SbVO₄ [7–10], Sb(VO₃)₃ [11] and Sb₂VO₅ [12]. Different from the rutile-type bulk counterpart, its nanocomposite structures strongly depend on their preparatory conditions. These oxides were found as catalysts for selective oxidation and ammoxidation of methane to formaldehyde [12], and propane to acrylonitrile [13]. In addition, nanoscale antimony vanadium also has potential electrochemical properties in the field of lithium ion batteries [7].

The nano-formed crystals can also be applied as coating materials to reach superhydrophobicity on even hydrophilic substrates. Such surfaces typically have high water contact angles (CA) more than 150° [14]. Different from wettability induced by conventional coatings from low surface energy materials (e.g., wax or oils) hydrophobic surface can also be ascribed to nano- to micro- meter scaled surface roughness [15–16]. Artificial superhydrophobic surfaces are generally

prepared by a two-step method [17]. Firstly, a rough surface is created by deposition of a micro- or nanomaterials, or etching operation [18]. Subsequently, molecules with a low surface energy will adhere to create water-repelling properties [19–20]. Chemical modification of surfaces with self-assembled monolayers, small molecules, polymer brushes, or physically changing the surface morphology, or combinations of both have been used to generate controllable wettability. Thus, printing SbVO₄ nanocrystals with tailored shapes onto the surfaces may lead to similar superhydrophobicity and benefit various domains such as drug delivery, micropatterning, self-cleaning coatings, anti-reflective, and oil removal, etc.

In spite of existing or potential applications of the antimony vanadium oxides, the synthesis conditions of these oxides are very harsh. Most of them need to be carried out under high temperature [6, 10, 11, 21–24]. The only wet-chemical synthesis through the hydrothermal method requires stringent conditions to reach high product purity [8]. Additionally, in contrast to its catalytic advantages, the wettability of the nano-formed SbVO₄ is scarcely reported. Therefore, it is necessary to explore new facile methods to control the properties and morphologies that will be eventually beneficial to multifunctional usages.

In this work, three different facile routes of hydrothermal method, reflux method and sonication method were employed to reach controllable morphologies of the SbVO₄ nanocrystals for practical superhydrophobic coatings. Instead of the NH₄VO₃ conventionally employed as the V source, the K₆V₁₀O₂₈·9H₂O is chosen in all synthesis processes, while pH values acts as the

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variable to control product properties. The drop-casting method was used to deposit nanoparticles onto the glass plate, followed by low surface energy fluorine silane to modify the properties of the superhydrophobic films. Static contact angle test results shows that all the antimony vanadium oxide films synthesized in our methods have excellent superhydrophobic properties and corrosion resistance. To the best of our knowledge, it is the first study of superhydrophobic properties of nano vanadium antimony. Besides, these simple, low-cost, environmentally friendly methods are also practical for large-scale nanoparticle productions.

Experimental

Materials

Antimonous chloride, 1H, 1H, 2H, 2H-perfluorodecyltriethoxysilane (PFOTS) were purchased commercially and used without further purification. $K_6V_{10}O_{28} \cdot 9H_2O$ was obtained from the laboratory. The glass substrate was obtained locally, which was cut into smaller 1.0 cm $\times$ 1.0 cm squares before usage. Then the glass was further cleaned by ultrasonic washing with acetone, anhydrous ethanol, deionized water for 15 min, respectively, and dried in a clean oven at 60°C for 1 h.

Preparation of SbVO₄

In our work, SbVO₄ nanoparticles are synthesized in three different procedures, including hydrothermal method, reflux method and ultrasonic method.

A common procedure was carried out to reach antimony and vanadium salt mixtures before wet chemical reactions. The SbCl₃ (0.912 g) was dissolved in HCl (4M, 8 mL) under vigorous stirring for 10 min. The $K_6V_{10}O_{28} \cdot 9H_2O$ (0.536 g) was dissolved in NaOH (4M, 8 mL) under vigorous stirring for 10 min. Afterwards, the two kinds of solutions were mixed in a slow dripping mode until gray precipitate turned up in the mixture. The solutions were stirred for another 10 minutes afterwards.

1. Hydrothermal method for preparation of SbVO₄ nanoparticles.

The pH value of the mixed solution was adjusted to 2.0, 4.0, 6.0 and 8.0 with 1 M HCl or NaOH solution. Then the mixture was stirred for 30 min at room temperature. The resulting mixture was transferred into a 45 mL Teflon-lined autoclave, maintained at 180 °C for 24 h, and subsequently cooled to room temperature. After natural cooling, the precipitates were collected and washed with distilled water and absolute ethanol for several times, and then dried in vacuum at about 60°C for 12h.

2. Reflux method for preparation of SbVO₄ nanoparticles.

In the reflux preparation, the resulting mixture was transferred into a round bottom flask and refluxed for 5 h at 100°C. The pH adjusting route, cooling, and washing steps were kept same as these in the hydrothermal synthesis.

3. Ultrasonic method for preparation of SbVO₄ nanoparticles.

In this synthesis, the mixture was stirred for 30 min at room temperature. The resulting mixture was transferred into ultrasonic cell disruptor and ultrasounded 30 minutes (200W) under room temperature. Other synthesis parameters and steps were kept the same as the previous.

Characterization

Powder X-ray diffraction (XRD) analysis was carried out on a MiniFlex II diffractometer with Cu K α radiation (continuous, 40 kV, 40 mA, increment = 0.02°, λ = 0.15406 nm) as the incident source. The morphology of as-prepared products was analyzed by the Scanning Electron Microscopy (SEM) (JEOL JSM- 6700F). The transmission electron microscope (TEM) images were recorded on a JEM-2010 microscope at an accelerating voltage of 200 kV. Chemical states of the elements in samples were deduced from the X-ray photoelectron

spectroscopy (XPS, Escalab 250, Thermo Scientific, USA). Contact angles were measured with Ramé-hart Model p/n 250-F1.

Preparation of superhydrophobic surfaces

Surface coatings were carried out via a facile drop-casting method. Typically, the as-prepared SbVO₄ samples were ultrasonically dispersed in ethanol to form a uniform suspension. Then several drops of the suspension were dripped on the glass substrate. When the ethanol was completely evaporated at ambient temperature, a thin film was formed on the surface of glass substrate. Finally, in order to reduce the surface energy of the fabricated structures, the film on glass substrate was modified by PFOTS.

Results and discussion

The X-Ray Power Diffraction (XRD) pattern of the as-synthesized SbVO₄ powder at 180°C for 24h under different pH value was shown in Fig. 1. As can be seen from the graph, all the diffraction peak data are basically consistent with the standard SbVO₄ (JCPDS:16 -0600, a = 4.598 Å, b = 4.598 Å, c = 3.078 Å), which belongs to the tetragonal phase. Especially at pH = 2.0 and pH = 8.0, the as-synthesized samples are almost completely corresponding to the tetragonal SbVO₄, which indicated that the prepared SbVO₄ had high purity. However, an obvious diffraction peak was detected in the image at the 2θ = 37.538°, and indexed to VO_{0.9} (111) plane. Under acidic condition, the intensity of the diffraction peak (2θ = 37.538°) increases with the pH value. When the pH value reached 6.0, the diffraction peak intensity is the strongest. This may be due to the fact that $H_2V_{10}O_{28}^{4-}$ captures a small amount of proton generated vanadium oxide under weak acidic conditions. When the pH value reached 8.0, the diffraction peak at 2θ = 37.538° disappeared due to the hydrolysis $V_{10}O_{28}^{6-}$ in alkaline environment. The best crystallization of the prepared SbVO₄ samples is obtained at pH = 8.0. But there is a small diffraction peak at 2θ = 29.9°, which is due to the formation of trace amounts of antimony oxide such as Sb₂VO₅ in excess of Sb³⁺ under alkaline conditions. All in all, we get a higher purity of SbVO₄ under strong acidic environment and weak alkaline environment. This shows that pH is very important for the synthesis of SbVO₄.

Fig. 1 XRD diagram of SbVO₄ prepared by hydrothermal method under different pH.

X-ray photoelectron spectroscopy (XPS) was used to elucidate the surface compositions and chemical states of the as-prepared SbVO₄. Figure 2a is the survey scan XPS spectrum, which clearly indicates that the samples of the SbVO₄ were mainly composed of Sb, V, and O elements. From Figure 7a, it is easy to conclude that there was no other prominent impure surfactant. The peak of C1s at 284.6 eV is from adventitious carbon in the instrument and used for calibration. Two peaks at 529.8 and 539.1 eV, are attributed to Sb3d_{5/2} and Sb3d_{3/2}, respectively [25, 26]. The characteristic signals of V⁵⁺ in

SbVO₄ were observed at 522.9 and 515.8 eV, which corresponds to V2p_{3/2} and V2p_{1/2}, respectively [27]. As shown in Fig. 2e, the peak at ~530 eV is assigned to the 1s XPS of the oxygen connected to the V–O bond [28]. In short, the results of XPS analysis are consistent with the result of XRD analysis.

Fig. 2 XPS spectra of SbVO₄ prepared by hydrothermal method (pH=8.0) (a) Survey of the sample (b) C1s (c) Sb3d (d) V2p (e) O1s.

Figure 3 shows the XRD pattern of the SbVO₄ powder synthesized by reflux reaction at different pH value. Similar to the previous analysis, all diffraction peak data are consistent with the standard tetragonal phased SbVO₄ (JCPDS:16 -0600, $a=4.598$ Å, $b=4.598$ Å, $c=3.078$ Å) patterns. From the figure, following the pH value, the diffraction peak intensity of samples is also notably changed, and gets sharpest at pH=4.0. The results show that the sample has good crystallinity at pH=4.0. In addition, there are few main diffraction peak of the sample in the XRD pattern, indicating the crystallization of samples in the whole reaction process is not very good. This is mainly due to the short reaction time and low temperature.

Fig. 3 XRD pattern of SbVO₄ prepared by reflux method under different pH value.

Fig. 4 displays the XRD pattern of the SbVO₄ powder synthesized by ultrasonic method (room temperature, 30min) under different pH value. From the figure, only one predominate peak turns up. The

diffraction peak data is consistent with the values from standard card (JCPDS:16 -0600, $a=4.598$ Å, $b=4.598$ Å, $c=3.078$ Å), belonging to the tetragonal structure of SbVO₄. The rather broad peak feature denotes either insufficient crystallization of the sample or dispersing of amorphous nanoparticles. This is mostly attributed to low reaction temperature and short reaction time.

Fig. 4 XRD pattern of SbVO₄ prepared by ultrasonic method under different pH value.

Crystallized mean sizes (CMS) of the as-prepared nanocrystals were calculated by applying the Scherrer formula to fit the (110) diffraction peaks in Fig. 1 (pH=8.0) and Fig. 3 (pH=4.0). The average diameters of SbVO₄ samples were estimated to ca. 24.74 nm, 9.78 nm, respectively. The calculated results of the Scherrer formula show that the SbVO₄ nanoparticles obtained by hydrothermal method are larger than those by the reflux method. This is mainly due to differences of wet-chemical conditions. In hydrothermal environments, sufficient temperature and reaction time contribute to the full reaction of Sb³⁺ and V₁₀O₂₈⁶⁻ to generate the SbVO₄ which can further grow to larger crystals. But in fact, after preliminary crystallization, the nanoparticles have higher dislocation density under hydrothermal conditions and therefore are not further fused to larger single crystalline nanoparticles. Instead, they agglomerated together in the form of low dimensional nanoparticles under high surface energy. On the other hand, the lower reaction temperature and shorter time in the other two methods prohibit large crystal formations. The as-formed nanocrystals are easier dispersed other than acting as centers for crystallizations.

Figure 5 depicts microscopic images of as-synthesized SbVO₄ samples (180 °C, 24 h) under different pH value during the hydrothermal process. From the Fig 5 (a) and (b), the samples have irregular block structure under low pH value, and the size is relatively large. When the pH value increase, the size of the sample become smaller. Figure 5 (c) shows the SEM image of the SbVO₄ prepared at pH=6.0. It can be seen from the figure that the sample is rather composed of aggregations of many small particles. The amalgamation trend further developed in the SbVO₄ prepared at pH=8.0 as shown in Fig.5 (d).

Discrepancies of the CMSs and nominal sizes of the nanoparticles are obvious. In Fig. 6, TEM results prove the bulks are mainly composed of many nanostructure with size of 50-100 nm. These nanostructures can further be divided to a large amount of smaller particles. Lattice determinations in HRTEM at Fig. 6 (c, e) verified that the particles are vanadium antimony nanocrystals with the sizes of 5-10 nm, and the interplanar spacing of 0.323 nm. This value corresponds to the (110) crystal plane, in agreement to the XRD results. The differences between CMSs and nominal particle size bear the growth processes of the products in wet conditions. Indeed, it's common that in the liquid synthesis, the products are firstly crystallized to nanocrystals which are further amalgamated to bigger

particles in sub-micro scales [29, 30]. TEM results also show that although obtained samples under different pH conditions displayed as block products under the SEM, but in fact the pH=8.0 sample is a kind of nanostructure where the surface roughness is much greater than that in low pH synthesis products. This is more advantageous to the superhydrophobic property of the product compared to block structures.

Fig.5 SEM of SbVO_4 prepared by hydrothermal method under different pH : (a) pH=2.0, (b) pH=4.0, (c) pH=6.0, (d) pH=8.0.

Fig.6 TEM and HRTEM under different magnification of SbVO_4 (pH=8.0 sample) prepared by hydrothermal method: (a, b) distribution of microstructures at low resolution, (c, d) Local magnified image of microstructure and the size distribution of SbVO_4 nanoparticles, (e) HRTEM of sample and (f) FFT image of local region in image (e).

Morphological results of SbVO_4 powders synthesized by reflux reaction (100°C , 5h) at different pH values were depicted in Fig. 7 (a)-(f), in turn for conditions with pH=2.0, pH=4.0, pH=6.0. Similar as these shown in Fig.5, the samples are composed of small nanocrystals. Figure 7 (b) and (c) also show a number of small nanoparticles, and these nanoparticles are well dispersed too. From the above analysis, it is found that the morphology of SbVO_4 samples prepared under different pH is the same. Thus, the reaction of pH has negligible impacts on the morphology of SbVO_4 samples,

but non-trivial on the crystallinity of the product. In order to clarify compositions of the product, TEM and HRTEM are also applied. By locally amplifying the nanoparticles in sample pH=4.0, we can see that the samples obtained by the reflux method are indeed composed of a large number of nanocrystals, which is obviously beneficial to the improvement of roughness of superhydrophobic films. And through the HRTEM figure 7 (e) and statistical chart of particle size distribution figure 7 (f), we can see that the size of the nanocrystals ranged from 3-7 nm and with the average diameter of 5.42 nm, which is slightly smaller than the calculations of the Scherrer formula in XRD pattern. The crystal spacing 0.323 corresponding to the (110) crystal plane of tetragonal SbVO_4 is also consistent with the results in XRD pattern.

Fig.7 SEM of SbVO_4 prepared by reflux method under different pH : (a) pH=2.0, (b) pH=4.0, (c) pH=6.0, (d, e) TEM and HRTEM of pH=4.0 sample, (f) and size distribution. The inset is the HRTEM and FFT image of pH=4.0 sample.

We also performed SEM determination for the morphologies of SbVO_4 samples prepared by ultrasonic reaction (30 min) under different pH value. As can be seen from figure 8, the morphology of the sample obtained by ultrasound is not significantly different from that obtained by the reflux method. The sizes of the SbVO_4 samples obtained under different pH are generally small. These nanoparticles are well dispersed. TEM pictures show that the size of the particles is about 50nm. However, the HRTEM shows that these nanoparticles are rather amorphous. The morphology of nanoparticles obtained by ultrasonic method is essentially different from that of the previous two methods. This is mainly due to the limitation of reaction temperature and time, the crystallinity of nanoparticles is much lower than those obtained by two other methods, but they also have greater coarseness, and the larger size distribution and amorphous state make it have lower surface energy, so they may be also favorable for super hydrophobic.

Fig.8 SEM of SbVO_4 prepared by ultrasonic method under different pH : (a) pH=2.0, (b) pH=4.0, (c) pH=6.0 and (d) TEM of sample pH=4.0. The inset is the HRTEM and FFT image of sample pH=4.0.

Through the above evidences, we found that samples are endowed with considerable roughness under the reflux and ultrasonic preparations in the acidic environment despite the limit crystallization. The nanostructures contain large amount of nanoparticles as a result of light aggregation. The Micro-nano structure not only has great roughness, but also can reduce the surface of the structure to a certain extent, which helps to reduce the wettability of material surface, and achieve super hydrophobic properties.

Fig. 9 shows the photograph of spherical water droplets on the surface treated with PFOTS. The wetting behavior can be interpreted by the Wenzel mode shown in eq. (1):

$$\cos \theta^* = r \cos \theta \quad (1)$$

Here, θ^* is the apparent contact angle, θ is the intrinsic contact angle, and r is the surface roughness factor, representing the ratio of the actual solid / liquid area to the apparent solid / liquid area of the rough surface.

It can be seen from eq. (1), as r is always larger than 1, surface roughness will enhance surface wettability. That is, (a) when $\theta < \pi/2$, θ^* decreases with the surface roughness and the surface becomes more hydrophilic; and (b) when $\theta > \pi/2$, θ^* increases with the surface roughness and the surface becomes more hydrophobic. When the contact behavior of droplets on the superhydrophobic surface follows the Wenzel model, the droplet does not necessarily roll even the angle of inclination of the surface is large enough.

Fig. 9 Wenzel state of SbVO_4 superhydrophobic surface. Scale bar: 2 mm.

In the following section, we analyze wettability of the glass substrates after being coated by the SbVO_4 synthesized through different methods and different conditions. Figure 10 shows the surface CAs of SbVO_4 nanoparticle film after the treatments with PFOTS. Figure 10 (a), (b), (c), (d) are the water drops on the surfaces with coating materials synthesized via hydrothermal methods under pH=2.0, 4.0, 6.0, 8.0, where water CAs were found

153.2°, 141.2°, 157.4° and 160.1°, respectively. It can be seen from figure 10(b), the sample contact angle is less than 150°, without superhydrophobic properties. This is due to rather sparse flakes of the sample (as shown in Fig. 5 (b)) that decreases the evenness and roughness in the micrometer scale. The samples prepared under other pH value all own good superhydrophobic properties, especially sample prepared at pH=8.0. Therefore, both chemical modification and surface morphology are very vital factors to the superhydrophobic properties.

Fig. 10 CA of water droplets on the SbVO_4 film prepared by method 1 under different pH: (a) pH=2.0, (b) pH=4.0, (c) pH=6.0, (d) pH=8.0.

In order to further study the acid and alkali resistance of SbVO_4 samples, the super-hydrophobicity of the SbVO_4 (prepared at pH=8.0) was measured with water CA by using water droplets of 10 μL with different pH values. Results were showed in Fig. 11. The CA of products changed only slightly with pH value of the droplets. Both under strong acid or alkali conditions, the CAs of SbVO_4 coated thin films are more than 158°. This indicates that the sample has good acid and alkali resistance property. When the solution pH was turned to 5.0, the hydrophobicity of the sample was the best, and the contact angle reached 162.3°.

Fig. 11 Water contact angles of the modified resulting surface (method 1) according to the pH of water droplet.

To further evaluate the corrosion resistance of the surfaces, we also measured the CA of 5 wt% NaCl aqueous solution on the superhydrophobic surfaces prepared at different pH value. As shown in Figure 11, the static CAs of the films coated with SbVO_4 prepared at pH=2.0, 4.0, 6.0, 8.0 were 150.4°, 141.2°, 156.6° and 160.9°, respectively. Except for the SbVO_4 sample prepared at pH=4.0, the SbVO_4 synthesized at other pH value are well tolerated, with the surface coated of SbVO_4 prepared at pH=8 has the best tolerance to 5 wt% NaCl aqueous solution.

Fig. 12 Tolerance of PFOTS modified SbVO_4 superhydrophobic surface (method 1) to 5wt% NaCl aqueous solution: (a) pH=2.0, (b) pH=4.0, (c) pH=6.0, (d) pH=8.0.

Figure 13 is a schematic diagram of the superhydrophobic surface the CAs of films encapsulated with SbVO_4 samples prepared by reflux method (100°C, 5h) at different pH. As can be seen from the figure, the static contact angle of the SbVO_4 superhydrophobic film prepared at pH=2, 4, 6 was 163.5°, 159.1°, and 162.1°, respectively. Experimental results show influence from preparation pH values on the hydrophobic properties is negligible. All static contact angles of all the surfaces are above 159° while the value tops with materials prepared at pH=2.0.

Fig. 13 The contact angle of the SbVO_4 prepared by method 2 under different pH: (a) pH=2.0, (b) pH=4.0, (c) pH=6.0.

The pH influences on water CAs are also performed to the films coated with materials synthesized through method 2. As is shown in figure 14, when the water drops pH in the range of 3-13, the static contact angle of all the SbVO_4 thin films keeps above 150°. This shows that our SbVO_4 films had good resistance to acid and alkali. In addition, the static CA of the SbVO_4 film increases with then pH value, reaches maximum at pH=7.0, but then decreases. However, the SbVO_4 films prepared at pH=6 showed an increasing trend in the pH=11 of water droplets, which indicated that the SbVO_4 films prepared by pH=6 had a good tolerance in alkaline environment.

Fig. 14 Water contact angles of the modified resulting surface (method 2) according to the pH of water droplet: (a) pH=2.0, (b) pH=4.0, (c) pH=6.0.

Tolerance tests were also performed for these sequence of films coated by samples prepared with method 2. The tests were performed under the same conditions as previously mentioned. CA

values are 150.4°, 141.2°, 156.6°, 160.9° for 5 wt% NaCl aqueous solutions with pH=2.0, 4.0, 6.0, 8.0 respectively. All the superhydrophobic films have good tolerance to 5 wt% NaCl aqueous solution, especially at pH=4, the static contact angle reaches 156.2°, which has the best tolerance to 5 wt% NaCl aqueous solution.

Fig. 15 Tolerance of SbVO_4 superhydrophobic surface (method 2) to 5wt% NaCl aqueous solution: (a) pH=2.0, (b) pH=4.0, (c) pH=6.0.

Fig. 16 The CAs of the prepared SbVO_4 by method 3 under different pH: (a) pH=2.0, (b) pH=4.0, (c) pH=6.0.

The same measurements for the CAs, pH dependent CAs and tolerances tests were performed for surfaces coated with SbVO_4 samples prepared by method 3. The water CAs were depicted in Fig. 16 (a)-(c) for these coated with nanocrystals prepared in different pH values, while pH dependent CAs in Fig. 17, and tolerance test results in Fig. 16. In Fig. 16, the water CAs of superhydrophobic film modified with PFOTS are 160.2°, 168.4°, 157.1°, respectively, with the one prepared at pH=4 owing the largest CA value. This is due to the higher roughness of samples prepared by ultrasonic method (see Fig. 7). Corrosive tests in Fig. 17 shows that all films are kept superhydrophobic despite of the ambient pH values. Coating material prepared at pH=4 leads to better hydrophobicity in general, compared with its peers prepared at other pH values. In Fig. 18, it is obvious that all films pass the tolerance tests and keep their superhydrophobicity under 5wt% NaCl conditions.

Fig. 17 Water contact angles of the modified resulting surface (method 3) according to the pH of water droplet: (a) pH=2.0, (b) pH=4.0, (c) pH=6.0.

Fig. 18 Tolerance of SbVO_4 superhydrophobic surface (method 3) to 5wt% NaCl aqueous solution: (a) pH=2.0, (b) pH=4.0, (c) pH=6.0.

Conclusions

In summary, we have prepared superhydrophobic films with excellent properties by simply coating SbVO₄ nanoparticles to glass plates. The SbVO₄ nanoparticles are synthesized by three different synthesis methods at different reaction condition in our work. The sample prepared by hydrothermal method own the best crystallinity compared to the other two methods, which may be due to the higher temperature required for the formation of vanadium antimony crystals. But the contact angle test shows that the crystallinity of the products have no obvious influence on the superhydrophobic properties of the films. In the view of synthesis, the pH value plays an important role to define the final properties of the products. All samples obtained by the three methods can be applied as coating materials to reach superhydrophobic properties and corrosion resistance in various conditions. Besides merits facilitating large-scale materials manufactory, the work also provides more possibilities for the synthesis of novel excellent superhydrophobic materials.

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