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Optimum calcination temperature for the synthesis of nanostructured alumina for energy storage heat management applications

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Abstract

Heat management for rechargeable energy systems such as lithium-ion batteries are of great performance and safety value. Optimization of solid additives to liquid cooling systems are a field of research for the advanced materials selection and characterization. While alumina showed proper traits to be used as additive in the LIB liquid cooling systems, nano-structuring of the particles entails promises for further improvement of the structural and heat dissipating capabilities of the ceramic oxide. Here a neat and efficient synthesis route is reported to obtain nanostructured alumina with a crystallite size around 100 nm. The effect of calcination temperature, ranging from 400 °C to 800 °C was studied on the microstructure of the synthesized Al₂O₃ nanopowder. It was established that the calcination temperature of 600 $^{\circ}\mathrm{C}$ may be considered as the optimum temperature for obtaining a porous nano-sized alumina with uniformly distributed particle size. This can be a proper candidate for heat management systems using a liquid combination such as water/Al₂O₃ for energy storage systems e.g. LIBs.

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1 Introduction

Lithium-ion batteries (LIB) are a promising storage technology due to performance advantages over competing battery chemistries. LIBs provide excellent cycle life stability over hundreds of charge/discharge cycles They also feature high volumetric and gravimetric energy density, meaning more energy can be stored per unit volume or weight compared to alternate battery technologies. Environmentally friendly and relatively low-cost raw materials are other advantages for these batteries. All in all, the characteristics have made LIBs the dominant battery technology for consumer electronics and electric vehicles and also a hot research topic within the electrochemical energy communities [1–5].

The propensity of Lithium-ion batteries (LIBs) to overheat poses a safety risk for large battery packs and can also compromise the performance of devices at high temperatures. Consequently, a significant focus in storage system research is to mitigate the effects of operations at high temperatures and to develop effective thermal management systems for LIBs [6–9].

However, the tendency for LIBs to overheat presents a safety concern for large battery packs as well as a threat to device performance at elevated temperatures. A serious research trend within storage systems is then to contain the consequences of elevated temperature operations as well as providing efficient heat management systems for the LIBs. Effective thermal management is thus critical for the mass commercialization of LIBs. Both too high and too low temperatures negatively affect the battery performance, and the high temperatures are more detrimental. Specifically, it is found that battery surface temperature is always the highest near the anode and then cathode while the center and margin are typically low-temperature zones. [4, 10-13].

Managing these thermal gradients across the battery cell is crucial. If allowed to overheat, lithium plating and electrolyte decomposition can cause catastrophic failure. At the same time, overcooling a LIB below optimal operating temperatures reduces power and energy capabilities. This is then of great importance to optimize the operating temperature of the LIB operation.

When evaluating the performance of thermal management additives and cooling systems for lithium-ion batteries, some key parameters need to be scrutinized as follows:

• Temperature change (ΔT in °C or K) which reflects how effectively the continuous liquid phase in the cooling method can regulate battery temperature during operation. Here a smaller temperature rise indicates more efficient heat removal.

- Heat transfer coefficient $(h \text{ in W/m}^2\text{K})$ that measures the rate of heat dissipation from the battery per unit surface area per degree of temperature difference. Higher h values correspond to improved heat transfer capability.
- Thermal conductivity (k in W/mK) that indicates how well heat propagates through the solid phase of additives in the cooling medium. Higher thermal conductivity leads to faster heat dissipation from the battery pack.
- Heat transfer enhancement efficiency (in percentage) which compares heat transfer capability of an additive-based nanofluid to the base fluid. Higher percentages equate to greater enhancement effect from the additive.
- Cooling power (W) is the total heat energy the cooling system can remove per unit time. Higher cooling power means superior temperature regulation.
- Cool down time (seconds): Time required to reduce battery temperature by a specified amount, for example from peak operation temp to safe storage levels. Shorter times naturally signal faster, and to some extent more uniform cool down.

Consequently, any thermal management techniques employed to control the heat in the battery system must meet acceptance standards that are determined by the parameters mentioned earlier.

There are three common methods for managing the thermal issues in lithium-ion batteries: air cooling, liquid cooling and the use of phase change materials. Air cooling is the most prevalent cooling technique in the heat management of LIBs due to its simple process and low cost. The lightweight, gaseous nature of air along with simple system design using fans, ducts or fins, make air cooling an economical option can be utilized. In practice, the higher heat transfer coefficient of forced convection systems generally makes them more suitable for high-power LIB packs. Natural air cooling can suit some low-to-medium energy density batteries.

Nonetheless, air cooling methods have drawbacks when applied to high energy density LIB designs required for long-range electric vehicles. The low heat capacity of air limits its ability to remove heat, while fans and ducts add parasitic losses that reduce overall system efficiency. This has motivated research into alternative advanced cooling methods like liquid cooling and phase change materials to achieve superior temperature control.

Liquid cooling circulates a coolant fluid through

thermal control loops directly integrated with the battery cells, offering orders of magnitude higher heat capacity compared to air. Meanwhile phase change materials utilize materials that melt at a desired temperature threshold, absorbing significant heat in the process. Intelligently integrating passive and active cooling methods tailored to battery design and application power demands provides a promising avenue for thermal management of LIBs from consumer electronics to electric vehicles.

This method depends on convective heat transfer away from the battery module and into the outgoing air stream. Both natural convection (heat rises from the warm battery surface into ambient air) and forced convection (fan-driven airflow over the batteries) can be used in this method. In practice, the higher heat transfer coefficient of forced convection systems generally makes them more suitable for high-power LIB packs. Natural air cooling can only suit some low-tomedium energy density batteries [11, 14–16].

Liquid cooling has also attracted significant interest in thermal management owing to its inherently higher thermal conductivity in comparison with air-cooling systems. That greatly assists with the scaling up of large systems. While working on convective heat transfer principles, liquid cooling method involves a more complicated configuration. However, a notable concern associated with this method is the potential risk of coolant leakage, which demands careful consideration due to its higher likelihood [4, 17–19].

Phase change material (PCM) is another method for heat management of LIBs. The main principle of this method is on the absorption/desorption of the energy in conjunction with phase transformation which is considered as a driving force to enhance the dissipation of the extra heat from the battery module. The main advantage of this method is that it does not need accessories such as fans, ducts and valves unlike its competing methods. This will translate into decreased operational cost and also lighter total weight of the system [14, 20].

Based on economic considerations and environmental issues, liquid cooling and the use of phase change materials are the most usable methods for LIB heat management systems.

The use of pure liquid is, however, normally uncommon in LIB cooling systems due to lack of withstanding properties at high temperature and degraded thermal conduction. Using appropriate additives can amend the performance and longevity of the abovementioned methods. Additives support the cooling system in regulating temperature by excessively absorbing heat energy generated within the battery cell structure. The addition of auxiliary solid agents such as light weight, conductive ceramics then can improve the functionality of liquid cooling streams like SiC/water slurry and Al_2O_3 /water [1, 4, 21]. Incorporating these agents in nanostructure with high thermal conductivity into the base fluid of liquid coolants can significantly improve heat transfer and cooling capacity. Similarly, doping phase change materials with high conductivity reinforcements allows them to absorb and dissipate heat during phase transitions more efficiently. Choosing suitable additives also widens the working temperature range and prevents degradation over repeated thermal cycling.

Optimizing these additives is key to enabling the cooling system to effectively maintain desired battery temperatures and cell performance over extended operation. Further research into novel additives compatible with battery chemistry and the cooling system helps to unlock superior combinations with ideal heat capacities and conductivities - resulting in reliable, durable and passive cooling solutions [1, 4, 21].

Nanostructured metal oxides such as AgO, Al_2O_3 and CuO have demonstrated potential as additives for thermal management materials of LIBs.

Silver oxide (AgO) nanofluids, in particular, have been theoretically shown to effectively decrease peak temperatures and temperature non-uniformity within LIBs through enhanced heat dissipation. The studies also showed that among candidate materials, nanosized AgO possesses superior cooling capabilities indicated by the lowest simulated battery temperature rises.

These are promising aspects for the use of nanoparticles in thermal regulation processes. On the other hand, most research in this area is theoretical, and additional experimental confirmation is needed before these proposals can be deemed practical for real-world applications [8, 11, 17].

While AgO nanoparticles demonstrate excellent heat transfer properties, the high cost of silver may limit nanofluid mass-synthesis. As one of the most valuable global metals, procuring adequate silver supply to produce AgO nano-coolants economically at scale presents challenges. Therefore the attention naturally will be paid to Al_2O_3 and CuO; where alumina has more efficient properties in controlling temperature and also simpler synthesis method than CuO [9,11,22]. Sefidan et al. [23], studied alumina nano-fluids thermal management capability and showed that they the temperature raise can be properly controlled for an LIB pack equipped with such cooling system.

Colangelo et al. [24], also suggested using Al_2O_3 nanoparticles for thermal management of LIBs due to their suitable thermal and physical properties while being low cost. Khaleduzzaman et al. [25], investigated the thermal characteristics for Al_2O_3 -water, CuO-water, and SiC-water nanofluids. They examined the heat transfer for the cooling fluid in steady, laminar conditions using an incompressible flow with constant heat flux in the channel. They demonstrated an improved heat flux and thus higher heat transfer rate when Al_2O_3 -water and CuO-Water nanofluid slurries were used.

Yetik et al. [26], reported a computational study to explore the effect of Al_2O_3 nanoparticles in LIBs liquid cooling system. Their model's output showed that the usage of Al_2O_3 can enhance the heat transfer efficiency and thus performance capability of LIBs.

Liu et al. [27], also used γ -Al₂O₃ as additives in different weight percent in the liquid cooling system for LIBs. Their results presented clear reduction of the battery module temperature and enhancement in the entire cooling performance. The results were improved when the weight percentage of γ -Al₂O₃ in the systems increased from 0.1% to 2%.

With all these progresses in the use of Al_2O_3 there still appears to be a hole for increasing the performance of the liquid cooling system enhanced with this additive. It is tentatively accepted that nanostructuring of the ceramic oxides such as Al_2O_3 can confer unique thermal transport properties that can significantly exceed values for bulk forms of the same composition. For example, nanostructuring introduces a high density of interfacial boundaries between adjoining grains or particles within the nanostructured Al₂O₃. These internal interfaces scatter heat-carrying phonons which impedes heat flow in the lattice and enhance external thermal conductivity. Higher surface area of nano-Al₂O₃ is also a positive point to motivate nano-structing of the Al_2O_3 . Bonding of the particles to adjoining surfaces is improved in the nano size.

Here the key points seem to lie within synthesis techniques and the optimization that experimentalists can bring about in the to synthesize nanoparticles of Al_2O_3 for the rechargeable batteries cooling systems.

The affecting parameters in the synthesis can be categorized to considerations and accuracy in the purity and preparation of precursors, acidity of the solutions, agitation and mixing rules and processing and calcination temperatures.

Calcination as the last material formation step play a crucial role in controlling nanoparticle morphology and size, the traits that directly dictate the heat dissipating characters. Determining the optimal calcination temperature will be then central in the current research. In this work we report a precipitation method for synthesizing of Al_2O_3 nanoparticles and the optimization its calcination temperature to form nanoparticles fitting as additives in liquid cooling heat management system for LIBs.

2 Experimental

2.1 Materials

In the experiment hydrated aluminum nitrate $(Al_2O_3(NO_3) \cdot 9H_2O)$ 99% was used as purchased. Methanol as solvent and sodium hydroxide (NaOH) were also used in pure form as precipitating agent and deionized water for washing step during centrifuging period.

2.2 Synthesis

First methanol was stirred on a heating magnetic stirrer (Alpha Silver) then hydrated aluminum nitrate was added into it while stirring. The mixing was continued until a transparent solution was achieved. Following this step, the precipitating agent (NaOH) was introduced gradually to solution by a manual syringe. During the addition of NaOH, extra solvent content was used to maintain the desired pH range. The addition of NaOH was kept on until the solution reached to pH value of 11. At this pH the solution was being stirred for 1 hour while Al(OH)₃ precipitated in the bottom of the beaker.

After one-hour colloid solution was centrifuged for 10 minutes with 3000 revolutions per minute and this step was repeated 3 times. At the end of every centrifuge step, the solution was washed with deionized water and then the next centrifuge step was carried out. Subsequent to the centrifuging step, a white paste was achieved was further dried with an OT140 oven in 200 °C for 2 hours. Afterwards, the dried paste was calcined in the furnace by a heating rate of 5 C/min from 400 °C to 800 °C for 2 hour and consequently cooled by the furnace default cooling program. The calcined powders were collected for physicochemical characterizations. Figure 1 shows the flowchart of synthesis route.

2.3 Characterization

For XRD analysis a PHILIPS PW1730 apparatus with copper source and step size of 0.05 degree was utilized. Two theta range was 10 to 90 degree. High Score Plus software was used to analyze the X-ray pattern.

A TESCAN VEGA/XMU model Scanning Electron Microscopy was used to obtain micrographs of the samples. The image analysis was based on backscatter electron mode of the SEM and then Energy Dispersive X-ray Spectroscopy (EDS) was performed for an elemental analysis of the powders. Open source software ImageJ was utilized for determination of the particle size distribution.



Fig. 1. Flowchart for the synthesis of Al_2O_3 nanoparticles via precipitation method.

3 Results and discussion

3.1 XRD analysis

In Figures 2 and 3 X-ray diffraction patterns for Al_2O_3 nanoparticles at different temperatures are shown. As it can be seen from Figure 2, from 500 to 700 °C there is hollow baseline in pattern which represent amorphous structure but after 700 °C the hollow baseline vanishes and extra peaks appear in two theta of 40 °C to 60 °C. The presence of nanoparticles in the sample can widen the peaks in comparison to bulk alumina. As the temperature increased, a double peak is observed at 32 °C for instance for the sample calcined at 800 °C as shown in Figure 3.

This double peak was transformed to a triple peak in the same angle also in 22 °C a peak at the highest calcination temperature. Some shifts can be seen for the same peak that decreased to 20 °C for the first appearance at elevated temperatures. Comparing with standard JCPDS cards from the software database, Al_2O_3 phases are identified as marked in the patterns. Gamma, Chi and kappa are alumina's phases that were found in the XRD patterns.

A general peak shift in some of the main peaks was observed in Figure 3a. There, chi alumina peaks are clearly shown near 65 °C. In Figure 3b for 800 C calcinated sample, however, this peak shifted to the left side and occurred near two tetha of 60°. Another observation in the results is the change in the intensity of some main peaks as a result of the alternating calcination temperatures. This can be attributed to the alteration in the preferential crystalline direction in the alumina particles formed by the calcination maximum temperature.

Based on Figures 2 and 3 and available data about alumina nanoparticles it can be conferred that appearing peaks in higher temperatures is due to the occurrence of high temperature chemical reactions that facilitated the formation of nanostructured aluminum oxides. Disappearing of hollow baseline in the XRD patterns is attributed to the crystallization process above 600 °C. The formation of alumina intermediate phases, gamma, kappa and chi, can be an indication of potential for the application of the samples as additives for heat management in LIBs. The potential stems from the fact that intermediate phases are thermodynamically favorable for energy absorption. The energy absorption can lead to the conversion of the intermediate phases to more stable ones. Such phase transformation does not necessarily dictate a significant volume change which can be negative to the heat management system of LIBs. The presented XRD patterns in 400 °C and 600 °C are in proper agreement with previously published work [1] for the same alumina structure except for minor peak shifts. It is believed that the progression of intermediate reactions may cause amorphous hollow in XRDs to justify the shift in peaks. The extra observed peaks can be also related to volatile phases such as sodium aluminate unreacted alumina hydroxide phases.



Fig. 2. XRD analysis results for calcination temperature: (a) 400 °C, (b) 500 °C (c) 600 °C.



Fig. 3. XRD analysis results for calcination temperature: (a) 700 °C, (b) 800 °C.

3.2 Crystallite Size Analysis

Analyzing the XRD patterns for the synthesized powder, one can check the existence of nanoscaled materials by studying the corresponding crystallite sizes. Debye-Scherrer method was used for the calculation of the crystallite sizes. The data for the crystallite size study is presented in Figure 4. It is seen that that the Al_2O_3 particles are in nanoscale and there is an optimum calcination temperature here. The crystallite sizes just under 500 nm have no meaningful differences for the samples calcined at 400 and 500 °C. It is thought that at 400 – 500 °C, the aluminum hydroxide precursor has likely not fully transformed to crystalline Al_2O_3 . Lower crystallinity means grain growth is not sufficiently promoted, limiting nanoparticle size reduction. As the calcination temperature rises to 600 °C, the conversion to gamma-phase alumina will have substantially progressed, allowing recrystallization into smaller, more stable crystal domains around 100 nm.

However, increasing temperature further to 700 °C and 800 °C initiates significant Ostwald ripening mechanisms. This sintering effect causes dissolution of smaller particles and redeposition onto larger ones, coarsening particle size above the optimal 100 nm desired size. Additionally, any residual carbon-containing species from the methanol solvent likely aid crystallite size control through site-specific particle nucleation up to 600 °C. But at higher 700 °C temperatures, they undergo exothermic reactions which eventually disrupts controlled particle growth.



Fig. 4. Comparison of crystallite sizes of $\rm Al_2O_3$ nanoparticles for different calcination temperatures.

3.3 SEM analysis

In Figures 5 to 9 SEM micrographs of the samples are shown. The synthesized particles have high surface area but probably due to suboptimal milling process, agglomerated particles can be seen. The presence of agglomeration is not in favor of thermal properties of Al_2O_3 nanoparticles and should be further improved.

The presence of nanoparticles are also vouched by the micrographs in Figures 5 to 9 in an agreement with the XRD results.

Using ImageJ software, the presentation of particle size and particle size distribution is done for the optimum sample via a histogram shown in Figure 10. It is seen that the frequency of the particles under 100 nm and specifically 50 to 70 nm are the highest among the analyzed images. While the human and calculation uncertainty cannot be ruled out, the difference with the results from Debye-Scherrer may be justified by the consideration of the fact that crystallite size can be larger than the average actual particles size, where a collection of particles can constitute a typical crystallite.



Fig. 5. SEM pictures from calcined powder at 400 °C.



Fig. 6. SEM pictures from calcined powder at $500\,^\circ\mathrm{C}.$



Fig. 7. SEM pictures from calcined powder at $600\,^\circ\mathrm{C}$.



Fig. 8. SEM pictures from calcined powder at $700\,^\circ\mathrm{C}$.



Fig. 9. SEM pictures from calcined powder at $800\,^\circ\mathrm{C}.$



Fig. 10. Particle size distribution analysis using ImageJ based on SEM image for 600 °C.

Elements distribution and frequency for the optimum sample are shown in Figures 11 and 12. As expected, aluminum and oxygen are the most abundant elements in the synthesized nanoparticles. The presence of silver in the results stems from the usage of silver as coating layer for the sample and has no interpretation in the compositional context. The sample shown in Figure 11a, possesses high sodium content which is attributed to improper mixing in preparation steps to be fixed in the upcoming experimental work.

After detecting nanostructured alumina phases in our microstructures we can deployment its properties for our additives which can be said higher surface area than bulk forms, high thermal conductivity and ability of absorbing heat.



Fig. 11. EDS analyze result after calcination of alumina powder: (a) 400, (b) 500.

3.4 FTIR analysis

In Figure 13 FTIR analysis for two calcined powder has been illustrated. Based on XRD and SEM results we selected 600 °C and 700 °C for analyzing then important points in diagram were perused. First aluminum and oxygen bonds (Al-O) have been detected which validated that Al_2O_3 phases have been synthesized in this experiment. Presence of H_2O wavenumber in pattern can be proved that transitive alumina phases existed in synthesized powder. Also another reason for this assertion is that all of instable phases of alumina which call them transitive have amount of water in their structures. By increasing temperature, it can be predicted that less water will exist in structures, in addition to it can be seen in diagram by decreasing of intensity. Hydroxide groups also detected, while they did not have same pattern. In the other words, the one near $2500 \,\mathrm{cm^{-1}}$ is relate to carboxyl group which produce due to reaction between methanol and sodium ion (Na⁺) during addition of NaOH into solution this reaction can produce carboxyl group. Another hydroxide bond in our result relate to compounds of between nitrate group and other ions in solution.



Fig. 12. EDS analyze result after calcination of alumina powder: (a) 600, (b) 700, (c) 800.



Fig. 13. FTIR analysis result of 600 and 700 calcination temperature.

Another points that can obtain from diagram is we have more littles peaks in high temperature patterns which can be relate to happening more interactions and more inter-products during calcination. Based on existing more peaks around Al–O bonds may be higher temperature for calcination can make this peaks to other Al–O bonds.

It is mentioned that aluminum metastable phases have H_2O bond in their structures it helps that our additives have better interactions with cooling fluid which is water although.

4 Conclusion

In this study, a precipitation method for synthesizing Al_2O_3 nanoparticles by precursor of 9 hydrated aluminum nitride got used in order to utilized for heat management systems of LIBs. After preparation of Al_2O_3 it was calcined in different temperatures and further structurally and morphologically analyzed.

The presence of phases gamma, kappa and chi alumina are eminent. This appears to make the alumina nanopowder promising for heat dissipation proposes with the capacity for heat sink while keeping an almost the same overall size of the powders.

Analyzing the samples calcined at 400, 500, 600, 700 and 800 °C, it was clear that the particle size for the 600 °C sample is the smallest around 100 nm. The calcination temperature of 600 °C appears to strike an ideal balance between activation energy barriers to initiate crystallization, particle ripening kinetics and maintenance of grain refining effects. That is enabling reliable synthesis of approximately 100 nm-sized alumina nanoparticles well suited as thermal additives.

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