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Visualization of the Agglomeration Process of Copper Particles Used in the Manufacture of Current-Carrying Cells of Lithium-Ion Batteries

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Conflicts of interest

The authors declare that there is no conflict of interest.

Abstract. The present study aims to conduct a comparative analysis of dispersion media used for the synthesis and storage of copper nanoparticles derived from copper formate, and to evaluate their suitability for application in current collectors. Particle characteristics in two media, namely ethyl alcohol and a mixture of alcohol and ethylene glycol, were investigated using dynamic light scattering, rheological analysis, and refractometry. The results demonstrate that the combined solvent system produces nanoparticles with a smaller average diameter (56.7 nm compared to 107.1 nm in pure alcohol) and a narrower size distribution, with 83.4% of particles falling within the 64–128 nm range. To visualise the data, particle size histograms were constructed, and the distributions were approximated using normal and Pearson distribution models. Experimental findings further indicate that the rate of particle agglomeration in the alcohol — ethylene glycol medium is approximately two times lower than in pure alcohol. On the basis of these results, the alcohol — ethylene glycol mixture can be recommended as a stabilising dispersion medium for the long-term storage of copper nanodispersions intended for use in current collector applications.

Keywords: lithium-ion battery, current collectors, copper nanoparticle dispersion, agglomeration, dynamic light scattering, normal distribution, Pearson distribution

Authors' contribution

Mityagin D.O. — experiment conducting, data processing, working with distribution, writing; *Koronnov A.A.* — data processing, visualization; *Agasieva S.V.* — data processing. All authors read and approved the final version of the article.

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Визуализация процесса агломерации частиц меди, используемых при изготовлении токосъемных элементов литий-ионных аккумуляторов

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Авторы заявляют об отсутствии конфликта интересов.

Аннотация. Цель исследования — сравнительный анализ дисперсионных сред для синтеза и хранения наночастиц меди, получаемых из формиата меди, с последующей оценкой их пригодности для использования в токосъемниках. Посредством применения методов динамического светорассеяния, реологии и рефрактометрии изучены характеристики частиц в средах «этиловый спирт» и «спирт + этиленгликоль». Показано, что использование комбинированной среды позволяет получать частицы с меньшим средним размером (56,7 нм против 107,1 нм) и более узким распределением (83,4 % частиц в диапазоне 64–128 нм). Для визуализации данных построены гистограммы и аппроксимированы законы распределения (нормальное и Пирсона). Экспериментально установлено, что скорость агломерации частиц в среде «спирт + этиленгликоль» в 2 раза ниже, чем в чистом спирте. На основании результатов сделан вывод о целесообразности применения смеси спирта с этиленгликолем в качестве стабилизирующей среды для долговременного хранения медной нанодисперсии.

Ключевые слова: системы накопления энергии, токосъемники, нанодисперсия меди, динамическое светорассеяние, нормальное распределение, распределение Пирсона, регрессия

Вклад авторов

Митягин Д.О. — проведение эксперимента, обработка данных, работа с распределением, написание текста; *Короннов А.А.* — обработка данных, визуализация; *Агасиева С.В.* — обработка данных. Все авторы ознакомлены с окончательной версией статьи и одобрили ее.

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Introduction

The rapid pace of industrial development necessitates accelerated growth in the energy sector. Modern information technologies are associated with substantial electricity consumption. Advanced approaches are therefore being employed to design highly efficient energy devices [1; 2]. At the same time, in addition to achieving high efficiency, it is essential to ensure the reliability of power systems and electrical equipment [3; 4]. The future utilization of available energy resources largely depends on the advancement of efficient energy storage systems. Currently, conventional methods for storing electricity generated by stationary power plants are complemented by a range of emerging technologies [5].

Ensuring the mobility of individual electric transport requires the development of batteries capable of storing sufficient energy for extended travel distances [6]. Existing market solutions can largely be regarded as interim measures. From a

contemporary standpoint, lithium-ion batteries represent the most promising technology for electrical energy storage. Numerous approaches focus on improving battery performance by modifying the electrochemical properties of the cathode material. In contrast, considerably less attention has been devoted to current collectors, which provide contact with the electrical network. A substantial portion of energy losses occurs during current collection, making this issue particularly significant in the operation of energy storage devices. One potential approach to reducing such losses is the use of highly dispersed copper in the manufacture of current collectors. Copper is characterized by high electrical conductivity, stable performance over a wide temperature range, and diamagnetic properties, which help minimize interference from electromagnetic fields during current collection. However, the properties of copper nanoparticles largely depend on the synthesis method employed. A key challenge in selecting an appropriate method lies in the limited understanding of the feasibility of obtaining particles of different sizes through dispersion-based synthesis techniques. This study seeks to predict the formation of particles of specific sizes produced via the synthesis of nanodispersions from copper formate and to visualize their agglomeration over time during storage.

1. Objective and Problem Statement

The objective of this study is to visualise the processes involved in the synthesis of highly dispersed copper particles from copper formate and their subsequent agglomeration, as well as to determine the theoretical size distribution of particles formed in different media. Such visualisation is necessary to assess the feasibility of using particles produced by this method in the manufacture of current collectors for lithium-ion batteries, based on their size characteristics.

To achieve this objective, the following tasks were undertaken:

1. To synthesise copper nanoparticles from copper formate in two media: ethyl alcohol and a mixture of ethyl alcohol and ethylene glycol;

2. To measure and compare particle sizes and their distributions in both media using dynamic light scattering;

3. To visualise particle size distributions by constructing histograms and fitting theoretical models (normal and Pearson distributions) to the experimental data;

4. To investigate the kinetics of copper particle agglomeration in both media over a 24-hour period;

5. To evaluate the stability of the resulting nanodispersions and assess the practical prospects of each medium based on the obtained results.

2. Theoretical Analysis

Key Challenges Associated with Current Collectors

The principal characteristics governing the current collection process are reliability, cost-effectiveness, and energy efficiency. Reliability implies the absence of damage to both the current collectors and the electrical network that could lead to device failure. From the perspective of cost-effectiveness, various manufacturing technologies incorporating advanced materials may be considered. The use of novel materials can extend the service life of the device while reducing production costs. The energy efficiency of current collection is determined by the properties of the current collector itself, particularly the material composition and the contact surface area.

The primary losses in the current collection process arise from incomplete contact with the electrical network, which is largely determined by the manufacturing characteristics of the current collector. First, materials with high electrical conductivity must be employed. Second, electrical energy transmission should be uniform across the entire contact surface. These requirements necessitate the use of dispersed material compositions [7; 8]. The small particle size ensures a high contact density, thereby reducing electrical losses during current collection [8–10]. A distinctive aspect of copper nanoparticle production is the ability to obtain particles up to 200 nm in size from

virtually any copper salt precursor. This flexibility enables the selection of synthesis methods based on the cost of source materials, resulting in significant economic advantages for the overall technological process. At the same time, challenges remain in the storage and transportation of copper nanoparticles due to their tendency to agglomerate [11–13].

3. Experiment Procedure

3.1. Method of Production

In this study, copper formate was employed as the precursor for the synthesis of copper nanoparticles [14]. One of the key reagents used in the preparation of copper formate is copper carbonate, which can be obtained by reacting an aqueous solution of copper sulfate pentahydrate ($CuSO_4 \times 5H_2O$) with an aqueous solution of sodium carbonate (Na_2CO_3). Equal volumes (50 ml) of the prepared solutions were mixed and subsequently placed in an ultrasonic bath to promote the reaction. The reaction between these components proceeds as follows:

$$CuSO_4 \times 5H_2O + Na_2CO_3 > \\ > Na_2SO_4 + CuCO_3 + 5H_2O. \quad (1)$$

The reaction yields copper carbonate, sodium sulfate, and water. To isolate the desired copper carbonate, the reaction mixture is filtered to remove sodium sulfate. Filtration is carried out using a Büchner funnel. Following filtration, concentrated formic acid is added portionwise to the copper carbonate with continuous stirring. The resulting mixture is then placed in an ultrasonic bath to ensure thorough mixing of the components. This reaction leads to the formation of blue copper formate:

$$CuCO_3 + 2HCOOH + H_2O > \\ > Cu(HCOO)_2 \times 2H_2O + CO_2. \quad (2)$$

The resulting formate is added to various media in order to prevent particle agglomeration. In this work, ethyl alcohol and a mixture of ethyl alcohol and ethylene glycol will be used as the media.

3.2. Method for Measuring Particle Size

Particle size measurements were carried out using the dynamic light scattering method with a Zetasizer Nano ZS spectrometer (Malvern Instruments). Solvent viscosity was determined by rotational rheometry using a Kinexus Pro rheometer. The refractive index was measured with an IRF-454B2M refractometer. The measured solvent parameters are presented in Table.

Solvent indicators

Solvent	Refractive index	Viscosity, MPa*s, at 25°C
Alcohol + ethylene glycol (anhydrous)	1.137	4.5
Alcohol	1.360	1.096

Source: by D.O. Mityagin, A.A. Koronnov.

4. Results and Discussion

The prepared copper formate sample was introduced into ethyl alcohol at a concentration of 0.25 g per 100 ml. Particle size measurements were conducted in this solution at a temperature of 15°C using the dynamic light scattering method. Each experiment was repeated at least five times to verify the measured particle size and to confirm the absence of agglomeration at the initial stage. The results indicate that this synthesis method predominantly produces copper particles with an average size of 107.1 nm. However, the resulting dispersion is not stable, as the presence of larger particles is observed. In the second experiment, a mixture of ethyl alcohol and ethylene glycol was used as the dispersion medium. Following the change of medium, the particle size remained within a comparable range; however, repeated measurements revealed no signs of agglomeration. In this case, the predominant particle size was 56.7 nm, which makes these particles suitable for potential application in the manufacture of current collectors. On this basis, it can be concluded that this medium is more appropriate for storing the resulting nanodispersion. The measurement results are presented in the combined graph in Figure 1.

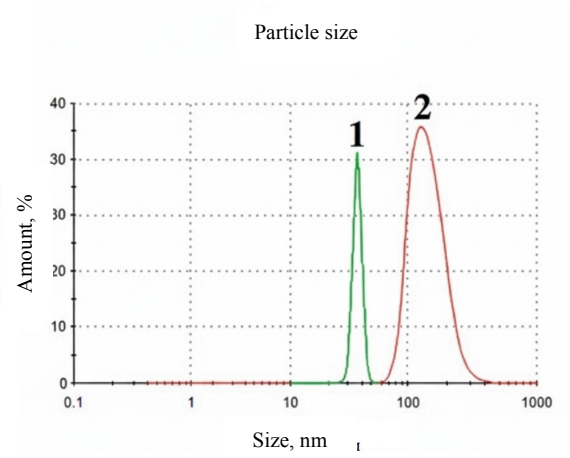

Figure 1. Measurement results using the Zetasizer instrument for two experiments:
1 — copper in the "alcohol + ethylene glycol" medium;
2 — copper in the "alcohol" medium
S o u r c e: by D.O. Mityagin, A.A. Koronnov.

Visualisation of the Distribution. For clarity, the experimental results are visualised [15; 16]. The data obtained from the graphs are processed to determine the percentage contribution of particles of different sizes to the total particle population. For this purpose, 20 data points are selected from each graph, and the proportion of particles corresponding to each measured size is calculated.

Histograms were constructed for both media to illustrate the particle size distributions. The data were grouped into class intervals (Figures 2 and 3): an interval width of 80 nm was selected for the alcohol medium, and 32 nm for the alcohol — ethylene glycol mixture. The histograms were then generated based on these grouped data. To further characterise the distributions, theoretical distribution functions were fitted in order to estimate the probability of occurrence of particles of different sizes. By truncating the dataset above 300 nm, attention can be focused on the central portion of the histogram, where a pronounced peak is observed. This feature justifies the use of a Gaussian distribution model for copper particles dispersed in alcohol. At the same time, the presence of an additional peak at lower particle sizes suggests the applicability of an alternative distribution function. For this type of data, the Pearson distribution was employed. The necessary parameters for both functions were calculated, and the corresponding curves were plotted.

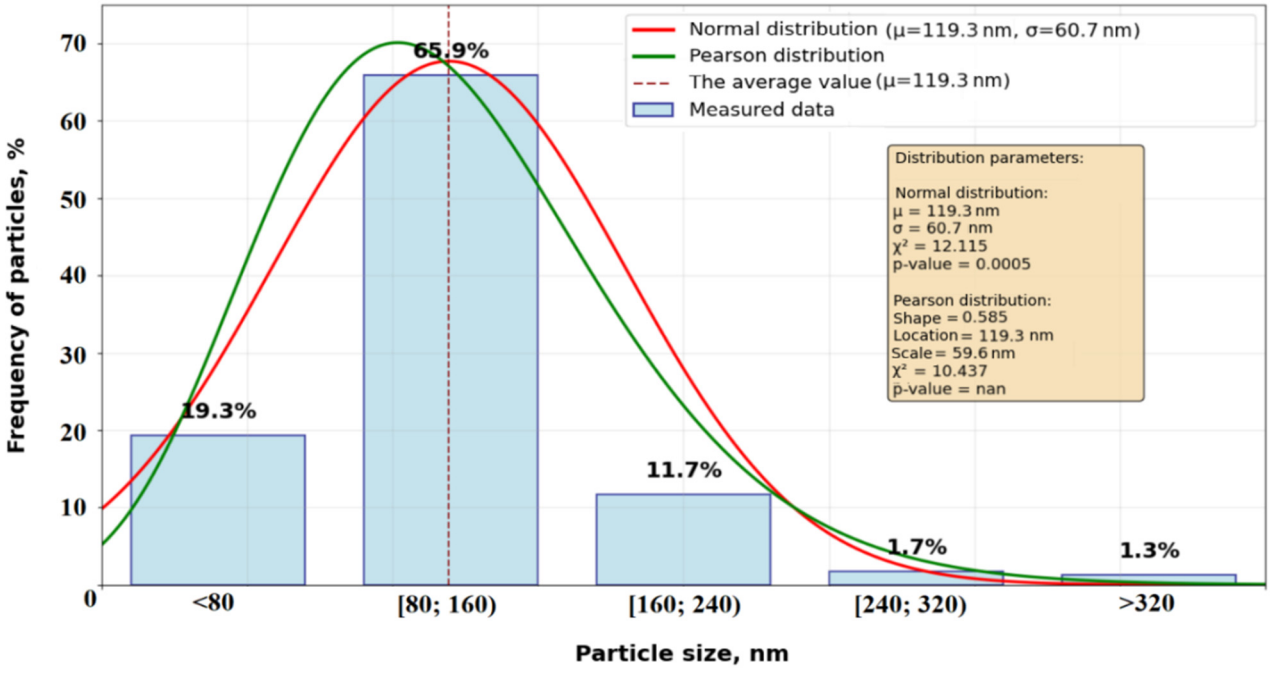

Figure 2. Gaussian distribution and Pearson distribution for particles in alcohol
S o u r c e: by D.O. Mityagin, A.A. Koronnov.

Based on the obtained distribution, it can be concluded that 65.9% of the particles fall within the size range of 80–160 nm. At the same time, the formation of larger particles in the dispersion is observed, which limits its suitability for the manufacture of current collectors. The presence of large particles in the subsequent thermal decomposition of copper formate would result in a reduced contact area and, consequently, increased energy losses during current collection.

Examination of the histogram for the "alcohol + ethylene glycol" medium reveals a pronounced peak at lower particle sizes followed by a gradual decline. After calculating the required parameters for the selected distribution function, the particle size distribution in the solution was plotted.

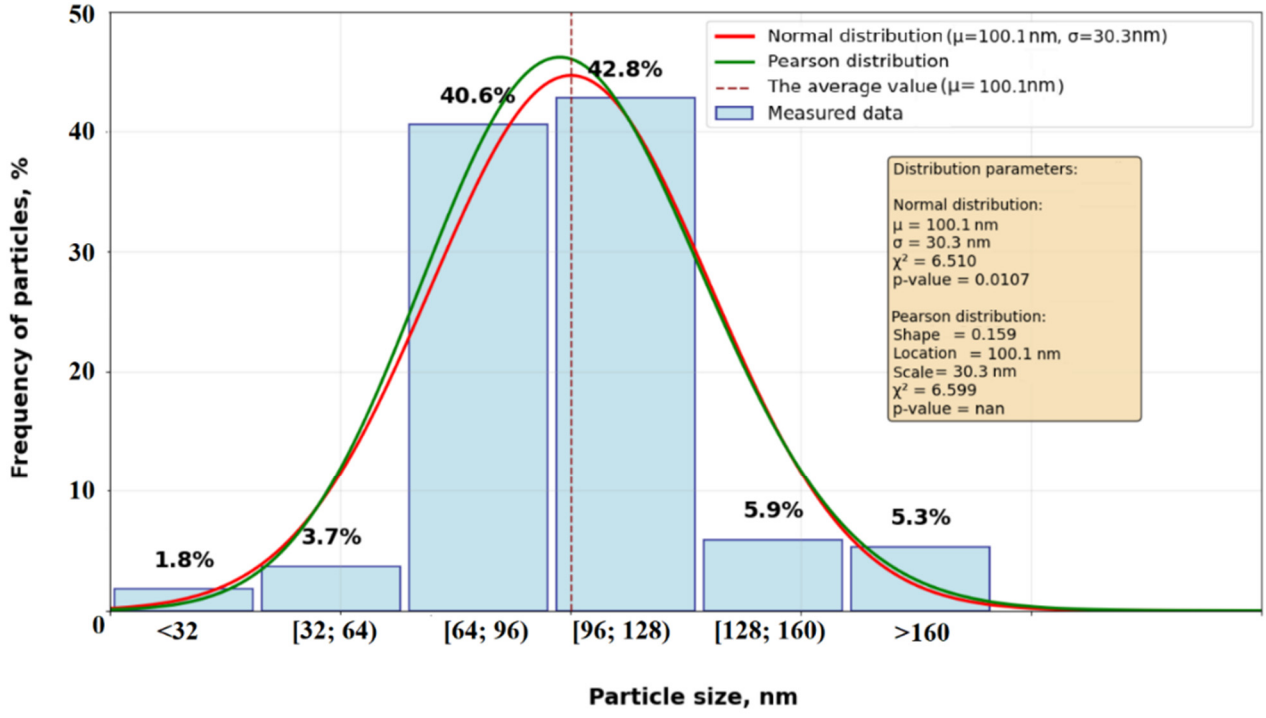

Figure 3. Gaussian and Pearson distributions for particles in a mixture of alcohol and ethylene glycol

S o u r c e: by D.O. Mityagin, A.A. Koronnov.

Based on the obtained distribution, it can be concluded that the majority of particles (83.4%) fall within the size range of 64–128 nm. The use of this medium therefore enables the production of particles suitable for the manufacture of current collectors. However, the presence of larger particles indicates a potential reduction in effective contact area. In this case, preliminary filtration may be applied, provided that the stability of the dispersion is maintained.

Over time, copper particles were observed to undergo agglomeration. Particle size measurements were performed every four hours over a 24-hour period. During this interval, the average particle size in the "alcohol" medium increased by a factor of 4.6, whereas in the "alcohol + ethylene glycol" medium it increased by 2.4 times. These results indicate that the alcohol — ethylene glycol mixture provides more favourable conditions for storing copper nanodispersions, as particle agglomeration proceeds at a slower rate.

The agglomeration process of copper particles over time in different media is illustrated below (Figure 4).

Analysis of the graph indicates that nanoparticles dispersed in pure alcohol exhibit low

stability. As a result, the use of this nanodispersion is associated with material losses not only during filtration but also due to ongoing agglomeration, since the presence of enlarged particles negatively affects performance in the manufacture of current collectors.

In contrast, nanoparticles in the "alcohol + ethylene glycol" medium demonstrate a slower rate of agglomeration. This improved stability makes it possible to retain the required parameters for current collector fabrication even after extended storage of the nanodispersion.

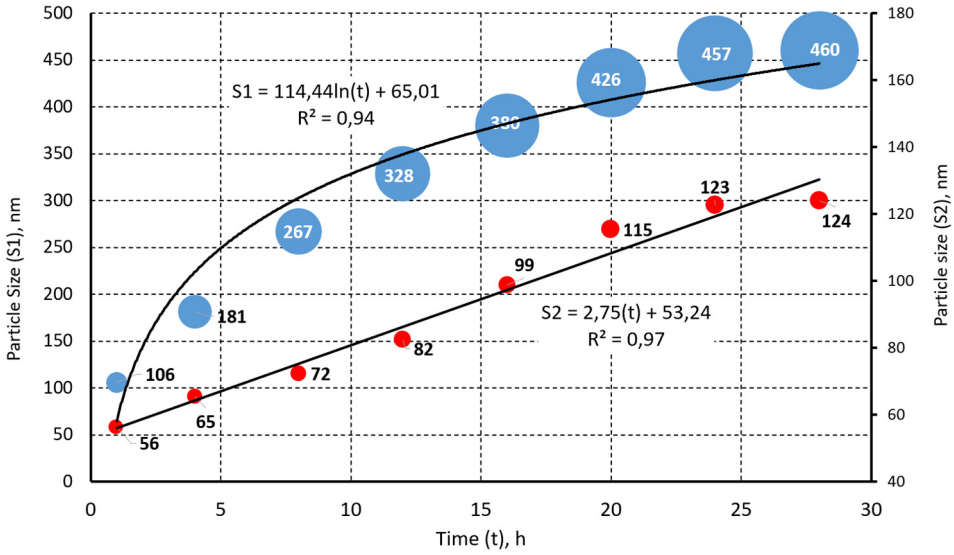

Figure 4. Copper agglomeration. S1 is the particle size in the "alcohol" medium, S2 is the particle size in the "alcohol + ethylene glycol" medium

S o u r c e: by D.O. Mityagin, A.A. Koronnov.

Conclusion

The study identifies the principal relationships describing the size distribution of copper particles in alcohol and in an alcohol — ethylene glycol medium, and examines the agglomeration process over a 24-hour period following synthesis. Histograms were constructed to characterise the particle size distribution and the proportion of particles of different sizes in each solution. In the alcohol medium, particles with an average size of approximately 107.1 nm are predominantly formed. In contrast, the alcohol — ethylene glycol medium yields smaller particles, with an average size of approximately 56.7 nm. The alcohol — ethylene glycol system demonstrates greater stability, as evidenced by a narrower particle size distribution (83.4% of particles within the 64–128 nm range). In pure alcohol, the distribution is broader (65.9% of particles within the 80–160 nm range), and larger particles are present that are unsuitable for practical application. Based on the results obtained, conclusions were drawn regarding the selection of dispersion media for producing copper particles of specific sizes, as well as for the storage of copper nanodispersions and their subsequent use.

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