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Scientific article

Electrospinning-assisted fabrication of ZnO nanostructures

A. Rakhmanova^{1*}, A. Jaldybayev², N. Khan¹, G. Yergaliuly³, A. Mentbayeva²,
B. Soltabayev^{1,3}

¹*Institute of Batteries, Astana, Kazakhstan*

²*Department of Chemical and Materials Engineering, School of Engineering and Digital Sciences, Nazarbayev University, Astana, Kazakhstan*

³*Advanced Sensors Laboratory, National Laboratory Astana, Nazarbayev University, Astana, Kazakhstan*

(E-mail: *aizhan.rakhmanova@alumni.nu.edu.kz, alikhan.jaldybayev@nu.edu.kz, natalya.khan@nu.edu.kz, gani.yergaliuly@nu.edu.kz, almagul.mentbayeva@nu.edu.kz, baktiyar.soltabayev@nu.edu.kz)

Abstract. The present study focuses on the synthesis of ZnO nanostructures via an electrospinning-assisted technique, followed by calcination under carefully controlled thermal conditions. The electrospinning process resulted in the formation of continuous, uniform, and bead-free polymeric precursor fibers, with a noticeable reduction in average fiber diameter. This clearly demonstrates the influence of electrospinning parameters, such as applied voltage, solution concentration, and flow rate, on the resulting fiber morphology. Subsequent calcination not only removed the polymeric matrix but also induced the crystallization of ZnO, transforming the fibrous structure into well-defined nanoparticles. The crystallite size of the obtained ZnO was strongly dependent on the calcination temperature, with the smallest crystallites achieved under optimized thermal treatment, thereby offering a route to fine-tune the nanostructure dimensions. Elemental analysis by energy-dispersive X-ray spectroscopy (EDS) confirmed the exclusive presence of zinc and oxygen, verifying the high purity and compositional integrity of the synthesized material. Furthermore, X-ray diffraction (XRD) analysis revealed sharp and intense diffraction peaks corresponding solely to the hexagonal wurtzite phase of ZnO, with no evidence of secondary phases or impurities. These results collectively demonstrate that electrospinning, coupled with controlled calcination, provides a robust, cost-effective, and scalable approach for producing high-quality ZnO nanostructures with desirable structural and compositional features. Such nanostructures hold significant promise for diverse applications, including gas sensing.

Keywords: ZnO nanoparticles, electrospinning, calcination, nanostructured materials, polymeric matrix, crystallite size, hexagonal wurtzite

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*the corresponding author

Introduction

Zinc oxide (ZnO) is a n-type semiconductor material that is suitable for a wide range of applications, including gas sensors, photocatalysts, solar cells, and optoelectronic devices. It has a high exciton binding energy of 60 meV and a wide bandgap of 3.37 eV [1]. Its significance in advanced functional materials has been further enhanced by its distinctive characteristics, including biocompatibility, nontoxicity, and high chemical stability [2]. Electrospinning has emerged as a straightforward, cost-effective, and highly controllable method for the production of one-dimensional (1D) nanofibers with high aspect ratios and large surface area-to-volume ratios, among the numerous synthesis techniques for ZnO nanostructures [3]. Applications such as gas sensing and catalysis are particularly advantageous due to the substantial enhancement of active surface sites by these structural features. A high-voltage electric field is applied to a polymer solution containing a Zn precursor during the electrospinning process, which results in the formation of continuous nanofibers. These fibers can be calcined to eliminate the polymer matrix and crystallize ZnO. Electrospinning provides superior control over fiber morphology, alignment, and porosity in comparison to other fabrication techniques, such as hydrothermal or sol-gel [4]. Furthermore, this method facilitates the incorporation of dopants or composite materials, which enables the final ZnO nanofibers to possess tunable physical and chemical properties. Consequently, electrospinning has emerged as the preferred method for the production of ZnO-based nanostructures for energy-related applications and advanced sensing [5].

The research offers the investigation of the nanosized ZnO via electrospinning method and subsequent calcination. Influencing factors, namely, calcination temperature, collector-to-tip distance, and voltage, are determined to control the particle size of ZnO.

The Literature review

In the past decade, electrospinning has been predominantly employed to produce nanoparticles from precursor solution containing natural and synthetic polymers with addition of metal salts. However, it has also been employed to produce ceramics, such as ZnO materials. For example, Liu et al. synthesized ZnO nanoparticles through electrospinning, followed by calcination, and analyzed their morphology, elementary composition, and crystal structure. There, it was specified that the resulted ZnO has a mesoporous nanofibrous structure which possess a single phase with favorable crystallinity [6]. Ultra-thin fibers of ZnO were prepared using electrospinning techniques, with poly(vinyl acetate) and zinc acetate as precursors. Characterization methods included thermogravimetric analysis, scanning electron microscopy, Fourier-transfer infrared, and X-ray diffraction [7]. The morphology and optical properties of zinc oxide fibres with diameters in the nanometre to micrometre range are reported by Viswanathamurthi. The PVA/zinc acetate organic/inorganic hybridnanofiberss were successfully prepared by electrospinning. Pure ZnO fibres were obtained by high-temperature calcination of the obtained fibers. Synthesised ZnO has a band gap of 3.13 eV and can be applied in different applications [8]. Electrospun ZnO was deposited on a glass substrate from zinc acetate dihydrate with polyvinyl acetate p olymer and annealed in the presence of oxygen until

organic molecules were decomposed. Characterisation results state that the mean fibre width was found to be 260 nm, and fibre thickness was measured at 460 nm. XRD patterns show that ZnO has a hexagonal wurtzite structure, and the material band gap for this electrospun ZnO fibre was found to be 3.28 eV. In summary, the n-type electrospun ZnO can be fabricated via facile electrospinning methods [9].

The methodology

PVP and ZnAc dihydrate were used as used precursors. 10 wt% solution of PVP in ethanol was made and stirred at room temperature for 5 h. Electrospinning was conducted at voltages of 14 kV and tip-to-collector distances of 10 cm. Nanofibers were collected on aluminum foil, which was then calcined in air at 600, 700 and 800 °C for 2 h to determine optimal sintering temperature. Selected parameters were applied to investigate the influence of on ZnO (Figure1). Scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDS), and X-ray diffraction (XRD) were employed to conduct a comprehensive analysis of the morphological and structural characteristics of the synthesized materials.

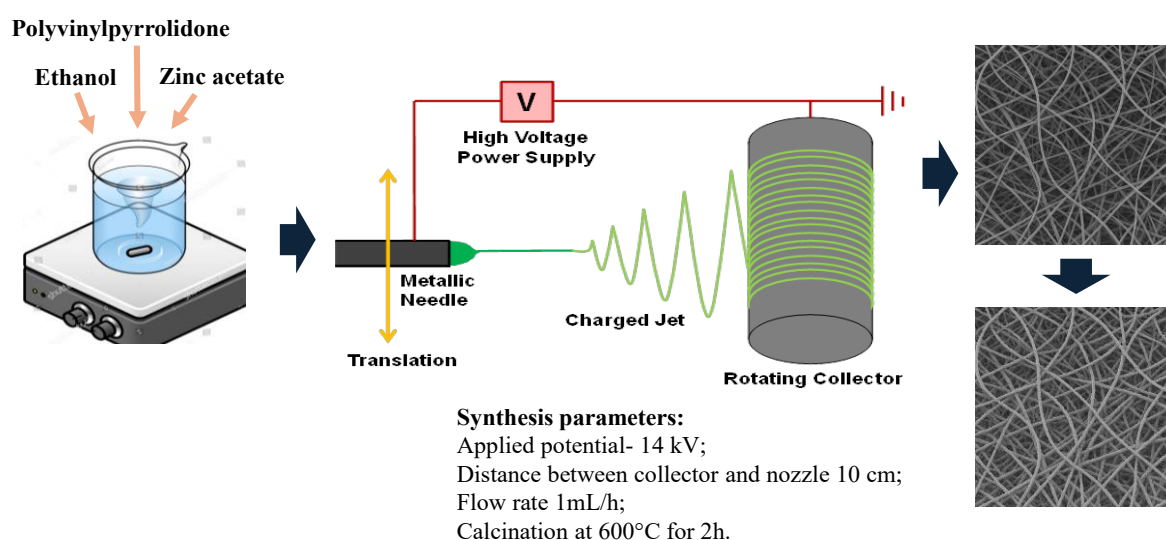


Figure 1 – Schematic illustration of synthesis procedure

Findings and Discussion

Effective electrospinning conditions were demonstrated by the formation of uniform, bead-free fibers across the samples, as indicated by SEM observations Figure 2a. The increased electrostatic force exerted on the polymer fluid at higher voltages, which enables the fiber to undergo greater stretching and elongation during electrospinning, resulting in a finer fiber morphology. In the same vein, the electric field intensity is increased by a shorter collector distance, which in turn promotes fiber thinning and uniform deposition [10]. The obtained fibers were effectively converted into ZnO particles with well-defined morphologies after

calcination Figure 2b. The crystallinity and particle size of ZnO are significantly influenced by thermal treatment. We observed a direct correlation between the calcination temperature and the size of the ZnO particles [7], [11]. Specifically, higher temperatures encourage grain growth and coalescence, while lower temperatures favour the formation of finer particles. In particular, the optimized electrospinning parameters are 14 kV voltage, 10 cm tip-to-collector distance, and 600 °C calcination resulted in ZnO particles with an average size of approximately 52 nm.

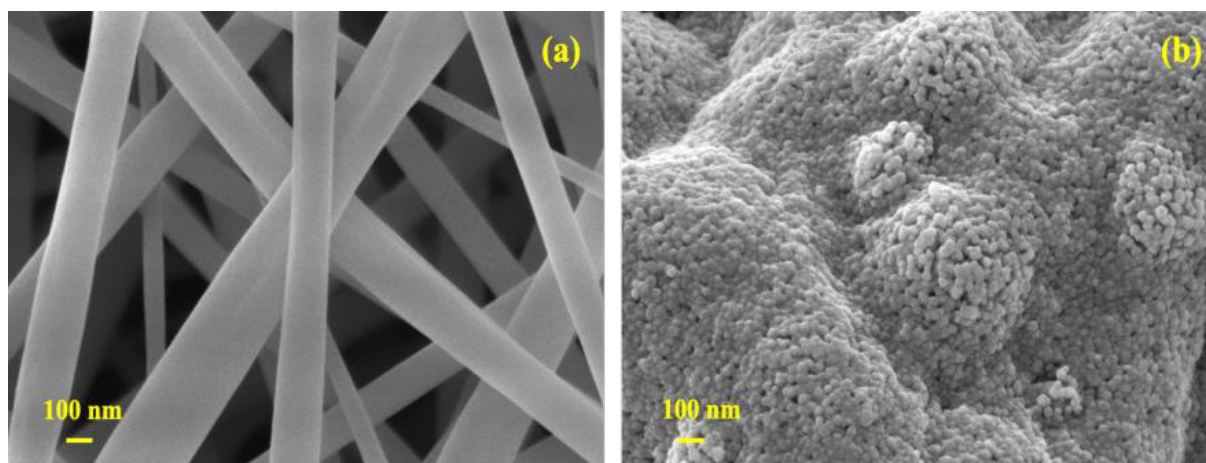
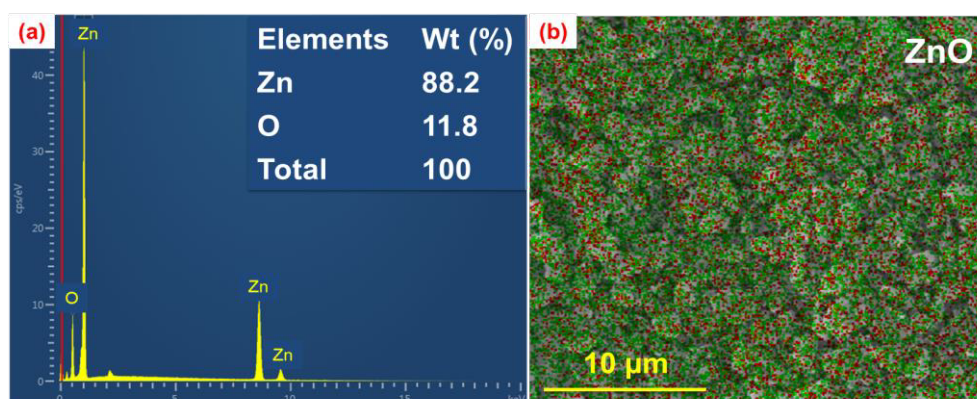


Figure 2 – SEM image (a) fabricated as spun fibers and (b) synthesized ZnO

The successful formation of ZnO was further validated by the Energy Dispersive X-ray Spectroscopy (EDS) analysis (Figure 3), which confirmed the presence of zinc (Zn) and oxygen (O) elements in their respective stoichiometric concentrations (Figure 3c and Figure 3d). The EDS spectra reveal that the synthesised material is of high purity, as there are no detectable impurity peaks. This suggests that the electrospinning and subsequent calcination procedure did not introduce other elements into the structure. The peaks corresponding to Zn and O are both strong and distinct. The formation of a well-ordered ZnO crystal lattice is also facilitated by the near-stoichiometric Zn:O ratio, which is crucial for the preservation of stable electronic properties and the improvement of the performance of ZnO-based devices, such as gas sensors [12], [13].



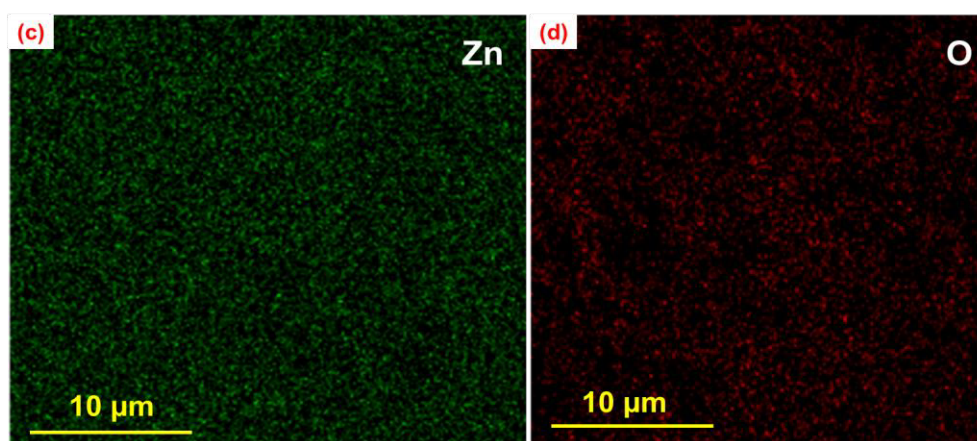


Figure 3 – EDS mapping of ZnO particles

The calcined samples exhibited a single-phase wurtzite structure of ZnO, as confirmed by X-ray Diffraction (XRD) analysis (Figure 4), without any detectable secondary phases or impurities. The hexagonal wurtzite crystalline structure is confirmed by the diffraction peaks observed at characteristic 2θ values, which correspond to the (100), (002), and (101) planes. The successful formation of phase-pure ZnO and the complete decomposition of the polymer matrix during calcination are indicated by the absence of additional peaks [1], [14], [15]. Therefore, among different calcining temperature 600 °C was determined as an optimal, as it produces clear, sharp, and intense diffraction peaks, indicating high crystallinity and phase purity of the material. At higher temperatures (700-800°C), a decrease in peak broadening is observed.

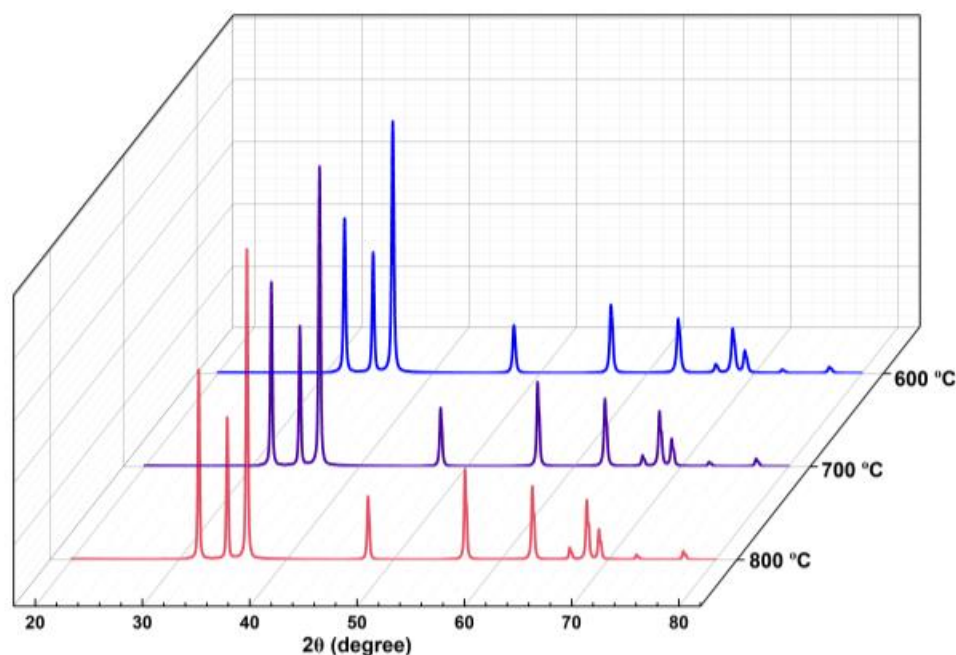


Figure 4 – XRD pattern of obtained ZnO nanoparticles

Conclusion

This research successfully synthesizes and controls ZnO nanoparticle size through electrospinning and calcination. Optimal synthetic conditions were reached for applied voltage, electrostatic distance, and calcining temperature. Optimal synthetic conditions under which uniform ZnO nanoparticles of 52 nm diameters in average could be achieved. This synthetic method allows morphology to be precisely controlled, and ZnO can be made compatible with sensor and electronic applications.

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Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Authorship contribution statement

A. Rakhmanova and **A. Jaldybayev** performed all the experiments, analyzed the data obtained and wrote the manuscript. Supervision, conceptualization, and revision were carried out by **N. Khan**, **G. Yergaliuly**, **B. Soltabayev**, and **A. Mentbayeva**. All authors have reviewed and approved the final manuscript.

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А. Рахманова^{1*}, А. Джалдыбаев², Н. Хан¹, Ғ. Ерғалиұлы³, А. Ментбаева², Б. Солтабаев^{1,3}

¹Аkkуmулятopлар институты, Астана, Қазақстан

²Химиялық және материалтану инженериясы кафедрасы, Инженерия және цифрлық ғылымдар мектебі, Назарбаев Университеті, Астана, Қазақстан

³Озық сенсорлар зертханасы, «Астана» Ұлттық зертханасы, Назарбаев Университеті, Астана, Қазақстан

(E-mail: *aizhan.rakhmanova@alumni.nu.edu.kz, alikhan.jaldybayev@nu.edu.kz, natalya.khan@nu.edu.kz, gani.yergaliuly@nu.edu.kz, almagul.mentbayeva@nu.edu.kz, baktiyar.soltabayev@nu.edu.kz)

Электроспиннинг әдісі арқылы ZnO наноқұрылымдарын алу

Андатпа. Бұл зерттеу қатаң бақыланатын термиялық жағдайда кейінгі қыздырумен бірге ZnO наноқұрылымдарын электроспиннинг әдісі арқылы синтездеуге арналған. Электроспиннинг процесі кезінде үздіксіз, біртекті және «түйіршіксіз» полимерлі прекурсорлы талшықтар алынып, олардың орташа диаметрінің айтарлықтай азаюы байқалды. Бұл қолданылған кернеу, ерітінді концентрациясы және берілу жылдамдығы сияқты электроспиннинг параметрлерінің талшық морфологиясының қалыптасуына ықпалын айқын көрсетеді. Кейінгі қыздыру тек полимерлі матрицаны жойып қана қоймай, сонымен қатар ZnO кристалдануын бастады, нәтижесінде талшықты құрылым жақсы қалыптасқан нанобөлшектерге айналды. Алынған

ZnO кристаллиттерінің өлшемі қыздыру температурасына айтарлықтай тәуелді болды: ең кіші мәндер оңтайлы термиялық режимде байқалды, бұл нанокұрылымдардың өлшемін дәл реттеуге мүмкіндік береді. Элементтік талдау (EDS) тек мырыш пен оттегінің болуын растады, бұл синтезделген материалдың жоғары тазалығын және құрамдық тұтастығын дәлелдейді. Сонымен қатар рентгеноструктуралық талдау (XRD) тек ZnO-ның гексагоналды вюрцит фазасына сәйкес келетін айқын әрі қарқынды дифракциялық шыңдарды көрсетті, бөгде фазалар мен қоспалардың ешқандай белгісі табылған жоқ. Жинақталған нәтижелер электроспиннинг пен бақыланатын қыздыруды біріктіру арқылы ZnO нанокұрылымдарын қажетті құрылымдық және құрамдық сипаттамаларымен алудың сенімді, үнемді және ауқымды тәсіл екенін дәлелдейді. Мұндай нанокұрылымдар әртүрлі қолданбаларда, соның ішінде газға сезімтал сенсорларда үлкен әлеуетке ие.

Түйін сөздер: ZnO нанобөлшектері, электроспиннинг, қыздыру (кальцинация), нанокұрылымды материалдар, полимерлі матрица, кристаллит өлшемі, гексагоналды вюрцит.

А. Рахманова^{1*}, А. Джалдыбаев², Н. Хан¹, Г. Ергалиулы³, А. Ментбаева², Б. Солтабаев^{1,3}

¹Институт аккумуляторов, Астана, Казахстан

²Департамент химической и материаловедческой инженерии, Школа инженерии и цифровых наук, Назарбаев Университет, Астана, Казахстан

³Лаборатория передовых сенсоров, Национальная лаборатория Астана, Назарбаев Университет, Астана, Казахстан

(E-mail: *aizhan.rakhmanova@alumni.nu.edu.kz, alikhan.jaldybayev@nu.edu.kz, natalya.khan@nu.edu.kz, gani.yergaliuly@nu.edu.kz, almagul.mentbayeva@nu.edu.kz, baktiyar.soltabayev@nu.edu.kz)

Электропрядение как метод получения наноструктур ZnO

Аннотация. Настоящее исследование посвящено синтезу наноструктур ZnO методом электропрядения с последующим прокаливанием при тщательно контролируемых термических условиях. В процессе электропрядения удалось получить непрерывные, однородные и свободные от дефектов типа «бусин» полимерные прекурсорные волокна, при этом наблюдалось заметное уменьшение среднего диаметра волокон. Это наглядно демонстрирует влияние параметров электропрядения, таких как приложенное напряжение, концентрация раствора и скорость подачи, на формирование морфологии волокон. Последующее прокаливание не только удалило полимерную матрицу, но и инициировало кристаллизацию ZnO, трансформировав волокнистую структуру в хорошо сформированные наночастицы. Размер кристаллитов полученного ZnO сильно зависел от температуры прокаливания: минимальные значения достигались при оптимальном термическом режиме, что открывает возможность точной настройки размеров наноструктур. Элементный анализ методом энергодисперсионной рентгеновской спектроскопии (EDS) подтвердил присутствие исключительно цинка и кислорода, что свидетельствует о высокой чистоте и целостности состава синтезированного материала. Дополнительно рентгеноструктурный анализ (XRD) показал резкие и интенсивные дифракционные пики, соответствующие исключительно гексагональной вюрцитной фазе ZnO, без признаков вторичных

фаз или примесей. Совокупность полученных результатов доказывает, что электроформование в сочетании с контролируемым прокаливанием представляет собой надёжный, экономичный и масштабируемый подход к получению высококачественных наноструктур ZnO с требуемыми структурными и композиционными характеристиками. Такие наноструктуры обладают значительным потенциалом для различных применений, включая газочувствительные сенсоры.

Ключевые слова: наночастицы ZnO, электропрядение, прокаливание, наноструктурные материалы, полимерная матрица, размер кристаллитов, гексагональный вюрцит.

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Information about the authors:

Aizhan Rakhmanova – the corresponding author, PhD, Senior Researcher at the Institute of Batteries, Nazarbayev University, Kabanbay batyr 53, Astana, Republic of Kazakhstan.

Alikhan Jaldybayev – Master student, Research assistant at the Department of Chemical and Materials Engineering, Nazarbayev University, Kabanbay batyr 53, Astana, Republic of Kazakhstan.

Natalya Khan – PhD, Senior Researcher at the National Laboratory Astana, Nazarbayev University, Institute of Batteries, Kabanbay batyr 53, Astana, Republic of Kazakhstan.

Gani Yergaliuly – PhD, Senior Researcher at the National Laboratory Astana, Nazarbayev University, Kabanbay batyr 53, Astana, Republic of Kazakhstan.

Almagul Mentbayeva – PhD, Associate Professor at the Department of Chemical and Materials Engineering, School of Engineering and Digital Science, Nazarbayev University, Kabanbay batyr 53, Astana, Republic of Kazakhstan.

Baktiyar Soltabayev – PhD, Head of Advanced Sensor Lab, Senior Researcher at the National Laboratory Astana, Nazarbayev University, Kabanbay batyr 53, Astana, Republic of Kazakhstan.

Айжан Рахманова – автор для корреспонденции, PhD, старший научный сотрудник Института аккумуляторов, Назарбаев Университет, просп. Кабанбай батыра 53, Астана, Республика Казахстан.

Алихан Джалдыбаев – магистрант, научный ассистент кафедры химической и материаловедческой инженерии, Назарбаев Университет, просп. Кабанбай батыра 53, Астана, Республика Казахстан.

Наталья Хан – PhD, старший научный сотрудник Национальной лаборатории Астана, Институт аккумуляторов, Назарбаев Университет, просп. Кабанбай батыра 53, Астана, Республика Казахстан.

Гани Ерғалиұлы – старший научный сотрудник Национальной лаборатории Астана, Назарбаев Университет, просп. Кабанбай батыра 53, Астана, Республика Казахстан.

Алмагуль Ментбаева – PhD, ассоциированный профессор кафедры химической и материаловедческой инженерии, Школа инженерии и цифровых наук, Назарбаев Университет, просп. Кабанбай батыра 53, Астана, Республика Казахстан.

Бактияр Солтабаев – PhD, руководитель Лаборатории передовых сенсоров, старший научный сотрудник Национальной лаборатории Астана, Назарбаев Университет, просп. Кабанбай батыра 53, Астана, Республика Казахстан.

Айжан Рахманова – хат-хабар авторы, PhD, Назарбаев Университеті, Аккумуляторлар институтының аға ғылыми қызметкері, Қабанбай батыр даңғылы 53, Астана, Қазақстан Республикасы.

Алихан Джалдыбаев – магистрант, Назарбаев Университеті, Химиялық және материалтану инженериясы кафедрасының ғылыми ассистенті, Қабанбай батыр даңғылы 53, Астана, Қазақстан Республикасы.

Наталья Хан – PhD, Назарбаев Университеті, «Астана» Ұлттық зертханасы, Аккумуляторлар институтының аға ғылыми қызметкері, Қабанбай батыр даңғылы 53, Астана, Қазақстан Республикасы.

Ғани Ерғалиұлы – PhD, Назарбаев Университеті, «Астана» Ұлттық зертханасы, аға ғылыми қызметкері, Қабанбай батыр даңғылы 53, Астана, Қазақстан Республикасы.

Алмагүл Ментбаева – PhD, Назарбаев Университеті, Инженерия және цифрлық ғылымдар мектебі, Химиялық және материалтану инженериясы кафедрасының қауымдастырылған профессоры, Қабанбай батыр даңғылы 53, Астана, Қазақстан Республикасы.

Бақтияр Солтабаев – PhD, Назарбаев Университеті, «Астана» Ұлттық зертханасы, Озық сенсорлар зертханасының жетекшісі, аға ғылыми қызметкер, Қабанбай батыр даңғылы 53, Астана, Қазақстан Республикасы.



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