



UDK: 541.64:539.211

Khusan YAKHSHINOROV,
PhD student at the National University of Uzbekistan
E-mail: husanyakhshinorov@gmail.com
Alibek ALIMOV,
Master's student of the National University of Uzbekistan
Urozboy MAMATOV,
Master's student of the National University of Uzbekistan
Elyor BERDIMURODOV,
Professor at the National University of Uzbekistan
Khamdam AKBAROV,
Professor at the National University of Uzbekistan
Jaykhun MAMATOV,
Associate Professor at the National University of Uzbekistan

Based on the review of Associate Professor N.Umirov from Qarshi State University

SYNTHESIS OF CARBON DOTS BASED ON CHITOSAN AND UREA

Annotation

Carbon dots (CDs) are one of the latest representatives of luminescent carbon nanomaterials. Due to their high solubility and stability, biotechnological compatibility, and low toxicity, carbon dots have attracted the attention of researchers. Their applications in various fields, such as sensors, photocatalysis, corrosion inhibitors, and solar panels, make them economically and technologically significant. In this study, carbon dots were synthesized using chitosan and urea through the hydrothermal method. The effects of pH, time, and temperature on the synthesis process were investigated. Particle size was thoroughly analyzed using the NKT-N9 Nano Particle Sizer and Transmission Electron Microscopy (TEM).

Key words: chitosan, urea, hydrothermal method, carbon dots, TEM, nano sizer.

СИНТЕЗ УГЛЕРОДНЫХ ТОЧЕК НА ОСНОВЕ ХИТОЗАНА И МОЧЕВИНЫ

Аннотация

Углеродные точки (CDs) являются одними из новейших представителей люминесцентных углеродных наноматериалов. Благодаря их высокой растворимости и стабильности, биотехнологической совместимости и низкой токсичности, углеродные точки привлекают внимание исследователей. Их применение в различных областях, таких как сенсоры, фотокатализ, ингибиторы коррозии и солнечные панели, делает их экономически и технологически значимыми. В данном исследовании углеродные точки были синтезированы из хитозана и мочевины с использованием гидротермального метода. Исследовано влияние pH, времени и температуры на процесс синтеза. Размер частиц был детально проанализирован с помощью наночастицемера NKT-N9 и трансмиссионной электронной микроскопии (TEM).

Ключевые слова: хитозан, мочевина, гидротермальный метод, углеродные точки, TEM, наноразмерный анализатор.

XITOZAN VA MOCHEVINA ASOSIDA UGLEROD NUQTALARINI SINTEZ QILISH

Annotatsiya

Uglerod nuqtalar (CDs) lyuminescent uglerod nanomateriallarining so'nggi vakillaridan biridir. Ularning yuqori eruvchanligi va barqarorligi, biotexnologik mosligi va past toksikligi tufayli uglerod nuqtalar tadqiqotchilarning e'tiborini tortmoqda. Sensorlar, fotokataliz, korroziya ingibitorlari va quyosh panellari kabi turli sohalaridagi qo'llanilishi ularni iqtisodiy va texnologik jihatdan muhim qiladi. Ushbu tadqiqotda uglerod nuqtalari xitozan va mochevinadan gidrotermal metod orqali sintez qilindi. Sintez jarayoniga pH, vaqti va haroratning ta'siri o'rganildi. Zarralar o'lchami NKT-N9 Nano zarralar o'lchagichi va transmissiya elektron mikroskopiyasi (TEM) yordamida batafsil tahlil qilindi.

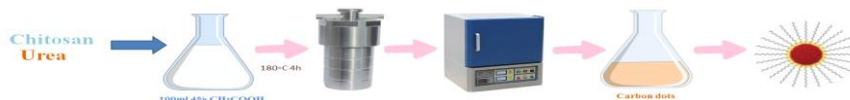
Kalit so'zlar: xitozan, mochevina, gidrotermal metod, uglerod nuqtalari, TEM, nano o'lchagich.

Introduction. Carbon dots (CDs) have attracted significant attention due to their unique ultraviolet fluorescence properties and outstanding performance in various applications, such as cellular labeling materials, corrosion inhibitors, and heavy metal sensors. CDs are characterized as zero-dimensional carbon nanoparticles with quasi-spherical shapes, optical activity, and particle sizes ranging from 1 to 100 nm. Furthermore, CDs stand out among most nanomaterials due to their unique properties, including biocompatibility, photoluminescence, chemical stability, enzyme-mimicking behavior, surface functionality, hydrophilicity, and simple synthesis methods. Numerous studies have explored the synthesis of CDs from various natural and waste materials, including chitosan, an aminopolysaccharide derived from marine waste with excellent potential as a carbon source [1-5]. Chitosan is obtained from chitin, a nitrogen-rich biopolymer found in crustaceans and insects. It is the second most abundant natural polymer after cellulose, with approximately 10^{10} – 10^{11} tons of chitin regenerated annually for chitosan production. Besides its economic and renewable nature, chitosan offers additional advantages, such as high nitrogen content and multiple functional groups, including acetamido, amino, and hydroxyl groups. Carbon dots derived from chitosan have been widely applied in various fields, such as corrosion inhibition in the chemical industry, pollutant detection, active food packaging, biosensing, and bioimaging. In this study,

carbon dots were synthesized using the hydrothermal method with chitosan and urea as precursor materials. The hydrothermal technique is one of the most commonly used methods for CD synthesis due to its affordability, eco-friendliness, and ease of controlling parameters such as temperature and reaction time. During the synthesis process, chitosan - alone or in combination with other precursors - is typically dissolved in water or acetic acid solution and transferred to a Teflon-lined stainless steel autoclave [6-9]. The stainless-steel reactor must be adequately sealed and heated at moderate temperatures (below 220°C) under autogenous pressure. After the reaction is completed and the system cools to room temperature, the obtained product is collected, separated, and purified. Researchers have reported various purification techniques, including filtration, centrifugation, and dialysis, or combinations of these methods. Finally, the resulting CD solution is either stored in a refrigerator or freeze-dried to obtain solid samples for further applications or characterization [10-15].

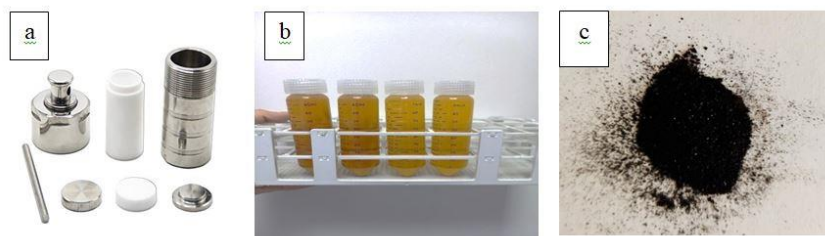
Required Reagents. chitosan, urea, sodium hydroxide, acetic acid

Synthesis: For the synthesis of carbon dots based on chitosan and urea, chitosan (95%, Yuwi Jinchi Cosmetics Co Ltd., China) is first dissolved in 100 mL of 4% CH₃COOH (acetic acid) solution. The chitosan is thoroughly dissolved, and an equal amount of urea is added to the solution. The pH of the solution is then adjusted to pH = 9 by adding 0.1N NaOH. The mixture is stirred for 15 minutes and then subjected to ultrasonication for an additional 15 minutes. Finally, the reaction is carried out using the **hydrothermal method at 180°C for 4 hours (1-picture).**



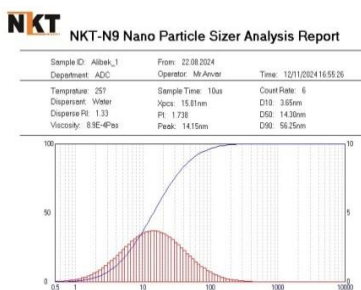
1-picture. Synthesis stages

After the reaction is complete, it is cooled to room temperature. The obtained chitosan and urea carbon dots are purified in several stages. Initially, they are centrifuged (13000 r/m for 15 minutes), filtered, and then dialyzed for 72 hours to obtain the carbon dots in a pure solution. In the next stage, they are dried at 40°C, and the carbon dots in dry form are obtained. The dry carbon dots are stored in the refrigerator (2-picture).



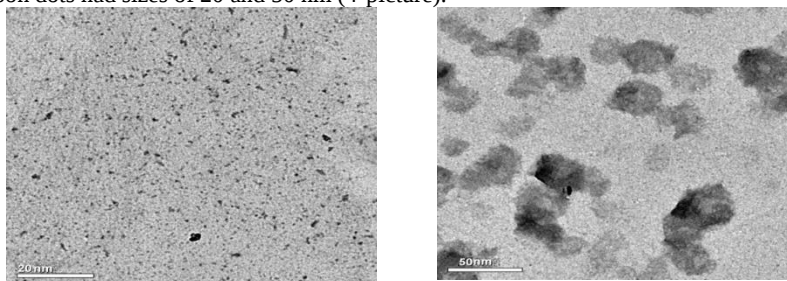
2-picture. Synthesis of carbon dots: (a) hydrothermal reactor, (b) carbon dots solution, (c) dry carbon dots.

Analysis of results. The best product was tested for particle size using the NKT-N9 Nano Particle Sizer, and it was found that the particle size ranged from 1 to 100 nm (3-picture).



3-picture. Analysis of chitosan urea-based carbon dots obtained using the NKT-N9 Nano Particle Sizer.

The size of the chitosan and urea-based carbon dots was examined using transmission electron microscopy (TEM). It was found that the obtained carbon dots had sizes of 20 and 50 nm (4-picture).



4-picture. Analysis of chitosan urea-based carbon dots obtained using transmission electron microscopy (TEM).

Conclusion. The particle size in the synthesis of carbon dots was found to be dependent on temperature and time. It was observed that 180°C for 4 hours provided optimal conditions and stability. By modifying chitosan with urea, the particle size was checked using TEM and found to be 20 and 50 nm. The obtained chitosan urea-based carbon dots are stable, inexpensive, environmentally friendly, non-toxic, and dissolve well in water.

REFERENCES

1. Saraswat V., Yadav M. Carbon dots as green corrosion inhibitor for mild steel in HCl solution // ChemistrySelect. - 2020. - №.5, №25. – P. 7347-7357.
2. Lv J., Fu L., Zeng B., Tang M., Li J. Synthesis and acidizing corrosion inhibition performance of N-doped carbon quantum dots// Russian Journal of Applied Chemistry. - 2019. - №. 92, № 6. - P. 848-856.
3. Cui M., Qiang Y., Wang W., Zhao H., Ren S. Microwave Synthesis of Ecofriendly Nitrogen Doped Carbon Dots for the Corrosion Inhibition of Q235 Carbon Steel in 0.1 M HCl // Int. J. Electrochem. Sci. - 2021. - №. 16. – P.151019-151020.
4. Q.H. Zhang, B.S. Hou, Y.Y. Li, G.Y. Zhu, H.F. Liu, G.A. Zhang, Two novel chitosan derivatives as high efficient eco-friendly inhibitors for the corrosion of MS in acidic solution, Corros. Sci. 164 (2020), 108346.
5. Cen H., Chen Z., Guo X. N, S co-doped carbon dots as effective corrosion inhibitor for carbon steel in CO₂-saturated 3.5% NaCl solution // Journal of the Taiwan Institute of Chemical Engineers. - 2019. -№. 99. - P. 224-238.
6. M.M. Solomon, H. Gerengi, Tugce Kaya, S.A. Umoren, Enhanced corrosion inhibition effect of chitosan for St37 in 15% H₂SO₄ environment by silver nanoparticles, Int. J.Biol. Macro. 104 (2017) 638 649.
7. A.M. Fekry, R.R. Mohamed, Acetyl thiourea chitosan as an eco-friendly inhibitor for mild steel in sulphuric acid medium, Electrochim. Acta 55 (2010) 1933–1939.
8. C. Zhou, Z. Li, J. Li, T. Yuan, B. Chen, X. Ma, D. Jiang, X. Luo, D. Chen, Y. Liu, Epoxy composite coating with excellent anticorrosion and self-healing performances based on multifunctional zeolitic imidazolate framework derived nanocontainers, Chem. Eng. J. 385 (2020), 123835.
9. C. Zhang, X. Luo, X. Pan, L. Liao, X. Wu, Y. Liu, Self-healing Li-Al layered double hydroxide conversion coating modified with aspartic acid for 6N01 Al alloy, Appl. Surf. Sci. 394 (2017) 275–281.
10. Cao S., Liu D., Wang T., Ma A., Liu C., Zhuang X., Ding H., Mamba B. B., Gui J. Nitrogen-doped carbon dots as high-effective inhibitors for carbon steel in acidic medium // Colloids and Surfaces A: Physicochemical and Engineering Aspects. - 2021. - №. 616. - P. 126280-126281.
11. Jain P., Patidar B., Bhawsar J. Potential of nanoparticles as a corrosion inhibitor: a review // Journal of Bio-and Tribo-Corrosion. - 2020. - №.6. –P.1-12.
12. Salleh S. Z., Yusoff A. H., Zakaria S. K., Taib M. A. A., Seman A. A., Masri M. N., Mohamad M., Mamat S., Sobri S. A., Ali A. Plant extracts as green corrosion inhibitor for ferrous metal alloys: A review // Journal of Cleaner Production. - 2021. - №. 304. –P. 127030-127031.
13. Haruna K., Saleh T. A., Quraishi M. A. Expired metformin drug as green corrosion inhibitor for simulated oil/gas well acidizing environment // Journal of Molecular Liquids. - 2020. - №. 315. –P. 113716-113717.
14. M. Zhang, L. Bai, W. Shang, W. Xie, H. Ma, Y. Fu, et al., Facile synthesis of water– soluble, highly fluorescent graphene quantum dots as a robust biological label for stem cells, J. Mater. Chem. 22 (15) (2012) 7461–7467.
15. X. Miao, D. Qu, D. Yang, B. Nie, Y. Zhao, H. Fan, et al., Synthesis of carbon dots with multiple color emission by controlled graphitization and surface functionalization, Adv. Mater. 30 (1) (2018) 1704740.