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Response Surface Methodology: A Versatile Tool for the Optimization of Particle Sizes of Cellulose Beads

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ABSTRACT

Synthesis parameters are of utmost importance for controlling the particle sizes of cellulose beads. This study aims to investigate the effects of synthesis parameters e.g., stirring speed (250–1250 rpm), surfactant concentrations (0.5–6.0% w/v), cellulose concentrations (1–5% w/v), and reaction temperature (30-100°C) on the particle sizes for micron-sized cellulose beads (μCBs) as well as other parameters e.g. the volume (1.0 mL) and concentration (0.1–1.0% w/v) of cellulose for nanosized (*n*CBs) cellulose beads using the response surface methodology (RSM). A total of 27 runs were conducted applying RSM based on the central composite design approach with Minitab-19. Cellulose concentrations were shown to have the most significant effect on both μCBs and *n*CBs. Under optimized conditions, the minimum and maximum mean particle size of μCBs that could be achieved were 15.3 µm and 91 µm, respectively. The predicted mean particle size for *n*CBs was obtained at 0.01 nm as the smallest and 200 nm as the biggest particle size under the optimum conditions. This study envisages that RSM and experiments for targeted applications such as biomedicine and agriculture could optimize the particle sizes of cellulose beads.

Keywords: Cellulose beads, controlled particle sizes, microbeads, nanobeads, response surface methodology

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INTRODUCTION

Cellulose fibers have attracted tremendous interest as a precursor material for the synthesis of micro-beads and nanobeads due to their vast availability, renewability, cost-effectiveness, biocompatibility,

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biodegradability (Balart et al., 2021; Gericke et al., 2013; Jampi et al., 2021; Kalia et al., 2011; Xu & Cho, 2022). Cellulose fibers can be isolated from various cellulosic wastes such as cotton, sawdust, and printed paper (Pang et al., 2011, 2018; Voon et al., 2016). Cellulose beads in nano and micron sizes have a high potential to be used in various technological and biomedical applications such as solid support, ion exchange, and water treatment (Alazab & Saleh, 2022; Saleh, 2021), protein immobilization (Califano et al., 2021; Culica et al., 2021; Guo et al., 2021) and delayed drug release (Gülsu & Yüksektepe, 2021; Ho et al., 2020; Mohan et al., 2022), slow-release fertilizer (França et al., 2021; Gomes et al., 2022; Machado et al., 2022), dye removal (Hamidon et al., 2022; Harada et al., 2021; Meng et al., 2019) and heavy metal removal (Du et al., 2018; Hu et al., 2018; Liu et al., 2021) due to their versatility and eco-friendliness (Carvalho et al., 2021).

Many researchers have attempted the water in oil microemulsion and nanoprecipitation methods to synthesize *μ*CBs and *n*CBs, respectively. Water in oil (W/O) microemulsion is thermodynamically more stable than oil in water by protecting the water-soluble molecules inside a continuous oil phase (Li et al., 2022; Russell-Jones & Himes, 2011; Song et al., 2020). Surfactant is crucially needed in forming microemulsion (Li et al., 2018). Owing to their adequate cutaneous tolerance, low irritation potential, and less toxicity than anionic surfactants, non-ionic surfactants are preferable, such as sorbitan monooleate, Span 80 as attributed to its biodegradability, biocompatibility, and safe to use in food, cosmetic as well as drug production (Conforti et al., 2021; Lechuga et al., 2016; Roque et al., 2020).

Nanoprecipitation is a straightforward and versatile method that involves the complex interaction between mixing, supersaturation, nucleation, and particle growth (Tay et al., 2012). Thanks to its simplicity and reproducibility, this process is greatly explored to synthesize nanoparticles (Yan et al., 2021). Response surface methodology (RSM) has been widely adopted as it helps achieve optimal conditions with a minimum number of trials (Karri et al., 2018; Sebeia et al., 2021; Shahnaz et al., 2020). Several studies have demonstrated that the RSM method is a very effective and versatile method for determining the effects of multiple synthesis parameters on the morphology and properties of the synthesized products (Allouss et al., 2019; Chin et al., 2021; Jancy et al., 2020; Lee & Patel, 2022; Pal et al., 2022; Wu & Hu, 2021).

The synthesis parameters profoundly affected the particle sizes of the *μ*CBs and *n*CBs (Chin et al., 2018; Voon et al., 2015, 2017b). However, without a proper understanding of the underlying mechanisms between each synthesis parameter and the particle sizes, it is very challenging to precisely and consistently control the particle sizes of the *μ*CBs and *n*CBs. Therefore, a systematic study of the effect of synthesis parameters on the particle sizes for *μ*CBs and *n*CBs by both the experimental method and the RSM is of utmost importance to allow precise control of their particle sizes for targeted applications. In this study, the effects of synthesis parameters on mean particle size for *μ*CBs and *n*CBs were investigated and optimized using the RSM.