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ORIGINAL PAPER

Taguchi approach-assisted optimization of spraying in non-solvent process for preparation of potassium perchlorate nanoparticles

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Abstract In this study, Taguchi robust design as a statistical route was utilized to optimize the variables of spraying in non-solvent process in order to achieve $KClO_4$ nanoparticles. The experimental factors of the spraying in non-solvent technique, which may be effective in $KClO_4$ particle size, were optimized efficiently via Taguchi method. The procedure operating conditions, i.e., $KClO_4$ concentration, solvent ratio, non-solvent identity, and spray steps, were considered at triple levels. The role of these operating conditions on the size of resulted $KClO_4$ particles was evaluated quantitatively by analysis of variance (ANOVA). It was found that the particle size of $KClO_4$ prepared by spraying in non-solvent technique might be adjusted efficiently by tuning the main parameters at the corresponding optimum level. Moreover, the optimal conditions for the preparing of $KClO_4$ nanoparticles via the studied method were proposed. The ANOVA exhibited that 3 % (w/v) as $KClO_4$ concentration, $CHCl_3$ as type of non-solvent, solvent to non-solvent ratio of 1:6, and single-step spraying are optimal conditions for the production of $KClO_4$ nanoparticles by spraying in non-solvent. Experimental data revealed that at optimum conditions of the process, the average particle size of produced $KClO_4$ is about 41 nm. Meantime, $KClO_4$ nanoparticles were prepared by supercritical carbon dioxide anti-solvent process for comparison. It was found that the size of the produced $KClO_4$ particles is about 55 nm.

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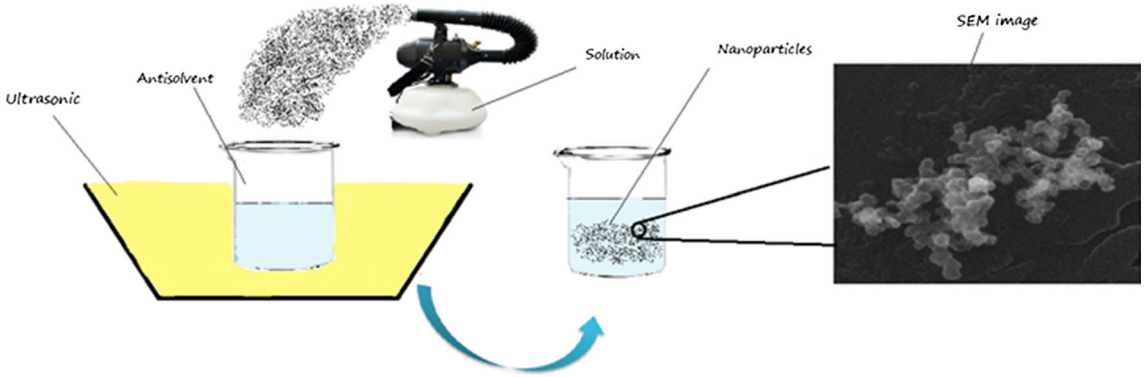
Graphical Abstract



Keywords Spraying in non-solvent process · Potassium perchlorate · Parameter optimization · Particle size

1 Introduction

Potassium perchlorate is widely used as an oxidizer in different energetic compositions, i.e., propellants and pyrotechnics [1–3]. The size and shape of potassium perchlorate particles have significant effects on the properties and performance of the propellants and explosive formulations [4–6]. Several properties of the energetic compositions, i.e., sensitivity, density, mechanical properties, total solid content, burning rate, and homogeneity of the formulation, are dependent on the particle size of the ingredients [7, 8]. The decrease in the size of this oxidizer and other ingredients leads to an increase in the reaction surface area, which could significantly enhance the combustion rate of the resulted energetic compositions [9–12]. In fact, nanoparticles of different materials commonly exhibit behaviors meaningfully different from those of the same material with the larger sizes [13–15]. The nanostructured materials due to their unique properties attracted a strategic concern in modern materials sciences.

During last years, excessive efforts have been focused on optimizing different routes to produce nanoparticles of different materials with well-defined properties [16, 17]. The spraying in non-solvent precipitation process has been widely used to crystallize different organic and inorganic materials in a wide range of sizes (micro and nano). This technique utilizes two liquid solvents that are completely miscible, while the solution of the compound is sprayed to the other solvents. In this technique, the solute to be micronized is soluble in the first solvent, but not soluble in the other solvents (non-solvent). In fact, spraying of the solute–solvent mixture to the second solvent induces the fine droplets of the solution to the second solvent and hence the formation of the supersaturated droplets in the second solvent and then precipitation of the solute as particles. Meantime, supercritical anti-solvent process (SAS) as a supercritical fluid nucleation route utilized carbon dioxide as an anti-solvent that causes precipitation of the substrate dissolved initially in a liquid solvent. Supercritical fluid-based techniques possess many advantages rather than the classical micronization processes, i.e., preventing thermal degradation of the sensitive target materials, no mechanical damaging, and no residual solvent in the resulted particles [18–20]. The method has been demonstrated in preparing a wide range of materials including polymers, dyes, and energetic materials [21–29].

Simultaneously, optimization routes, i.e., Taguchi experimental design, are included in previous planned array experiments and then their results are collected, pointing out the optimum conditions via retention mapping or by creating a response surface [30]. Taguchi robust design used the previously designed orthogonal arrays to hand over intended factors whose results might then be analyzed by a communal mathematical process [30–32]. Furthermore, the role of each factor on the investigated process might be independently figured. In other words, Taguchi method has this capability to separate the importance of different factors, by the aid of analysis of variance on the resulted data during performing the experiments according to the orthogonal arrays [33–37].

The main aim of this work was preparation of potassium perchlorate nanoparticles via spraying in non-solvent process. Thus, Taguchi robust design was utilized to optimize the variables of spraying in non-solvent process and evaluate the role of these factors on the definition of the particle size of resulted $KClO_4$. The factors included in this study were as follows: $KClO_4$ concentration, solvent to non-solvent ratio, non-solvent identify, and spraying steps. The produced $KClO_4$ particles at optimum condition of the

Table 1 Assignment of the factors and levels of the process using an OA_9 (3^4) matrix and average particle size of produced $KClO_4$ as a response

Experiment number	Anti-solvent	Concentration of $KClO_4$	Spray steps	Solvents ratio	Particle size (nm)
1	BuAc	1	1	1:3	630
2	BuAc	3	2	1:6	460
3	BuAc	6	4	1:9	780
4	$CHCl_3$	1	2	1:9	265
5	$CHCl_3$	3	4	1:3	160
6	$CHCl_3$	6	1	1:6	205
7	CH_2Cl_2	1	4	1:6	165
8	CH_2Cl_2	3	1	1:9	270
9	CH_2Cl_2	6	2	1:3	385

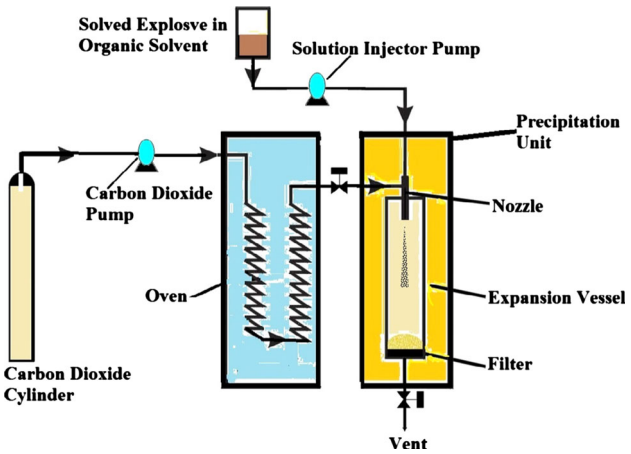


Fig. 1 Schematic of the experimental setup used for SAS process

spraying in non-solvent process were compared to the resulted SAS procedure.

2 Experimental

2.1 Spraying in non-solvent process precipitation process

The possibility of the formation of potassium perchlorate ($KClO_4$) nanoparticles was examined via spraying in non-solvent precipitation process. The chemicals used, including potassium perchlorate powder, dimethyl formamide, butyl acetate, chloroform, and dichloromethane, were all analytical grade from the Merck Company (Germany) and utilized as received. All experiments were conducted in an ultrasonic bath. The ultrasonic apparatus Elma D 78224 made in Germany was used. The experimental conditions of the spraying in non-solvent process for the micronization of $KClO_4$ were optimized by the aid of Taguchi experiment design approach. The studied operation parameters included, $KClO_4$ concentration, solvent for non-solvent ratio, non-solvent identify, and spraying steps. These factors were investigated at three levels via a L_9 array proposed by the Taguchi approach as shown in Table 1.

2.2 Supercritical fluid CO_2 anti-solvent process (SAS)

In the SAS process, the potassium perchlorate particles were dissolved in dimethyl formamide and then a carbon dioxide supercritical fluid, which is miscible with the liquid, but in lower solvent power to the potassium perchlorate, is added to recrystallize this oxidant. Carbon dioxide (99.99 % purity), in a cylinder equipped with an eductor tube, was from Sabalan Co. (Tehran, Iran). The scheme of the SAS system utilized in this work is given in Fig. 1. As shown in this figure, a heat exchanger is employed for adjusting the temperature of the CO_2 after compressing via the pump, while the pressure is controlled by the back-pressure-regulating valve. The utilized SAS system is consisting of a precipitation chamber, and carbon dioxide is entered into the precipitation chamber, after its pre-heating in the heat exchanger. Concurrently, $KClO_4$ solution is pumped, heated and fed into the precipitation chamber through the stainless steel nozzle (with 150 μm inner diameter). The nozzle is located at the top of the chamber which is located beside the inlet point of CO_2. A stainless steel filter possessing 200 nm pore size is located at the bottom of the chamber to collect the micronized particles, while letting them pass through the SC-CO_2/organic solvent mixture. Meantime, to control the flow rate of the leaving mixture, a valve was used next to the precipitator. The operating conditions of the SAS process for the preparation of potassium perchlorate ($KClO_4$) nanoparticles are presented in Table 2. These conditions were chosen based on the previous similar reports [32–34] and considering the accessible experimental conditions.

Table 2 ANOVA results for the micronization procedure of $KClO_4$ via solvent/anti-solvent procedure using OA9 (3^4) matrix

Pooled[f]								
Factor	DOF[a]	S^b	V^c	DOF*	S^*	V^*	F^d	P^e
Anti-solvent identification	2	297355.496	1486877.748	2	297355.49	148677.748	53094.28	78.583 %
Spray steps	2	5.6	2.8	–	–	–	–	–
$KClO_4$ concentration	2	39488.864	19744.432	2	39488.864	19744.432	7050.93	10.434 %
Solvent ratio	2	41538.874	20769.437	2	378388.88	20769.43	7416.96	10.976 %
Error	0	0	0	2	5.65	2.82	–	0.007

[a] DOF is degree of freedom for each variable

[b] S is the standard deviation (mean squares) for each parameter

[c] V is mean square (variance) value for each parameter

[d] F is the variance ratio or F statistic (the ratio of variance due to the effect of a parameter and variance value due to the error term)

[e] P is the percent contribution of each parameter or error term in the results of process

[f] The asterisked letters represent their corresponding terms after pooling of insignificant parameters at 90 % confidence level; pooled error results from pooling

2.3 Characterization of the micronized samples

The micronized $KClO_4$ samples via different experimental conditions spraying in non-solvent process and SAS process were characterized by scanning electron microscopy (SEM). The scanning electron micrographs were recorded on a Philips XL30 series instrument by employing a golden film to load the dried sample particles on the instrument. Golden films were obtained on a sputter coater model SCD005 manufactured by BAL-TEC (Switzerland). Transmission electron microscope (TEM) image was obtained on a Zeiss EM10C transmission electron microscope. The sample preparation was performed by coating on formvar carbon-coated grid Cu Mesh 300 before the measurement. XRD analysis was carried out on a Rigaku D/max 2500 V diffractometer equipped with a graphite monochromator and a Cu target.

3 Results and discussion

3.1 Optimization of spraying in non-solvent process parameters

In this research, the effect of various procedure parameters, i.e., $KClO_4$ concentration, solvent to non-solvent ratio, non-solvent identify, and spraying steps, on the size of the resulted $KClO_4$ particles was studied. Table 1 presents the examined factors and their tested levels. In Fig. 2, the SEM images of four $KClO_4$ samples which resulted in different conditions of the spraying in non-solvent process are given. On the other hand, the average size of resulted $KClO_4$ particles at each trial of Table 1 is given in the last column of this table as the response. As shown in Fig. 2 and Table 1, the size of resulted potassium perchlorate varies considerably and is depending on the operating condition of the process.

In Taguchi approach, analysis of the collected data, while there is no considerable interaction between examining factors, includes the following three steps: identification of the optimal conditions for the examined process; assignment of the singular effect of each factor on the process result (the average size of resulted $KClO_4$ particles); and then estimation of the process efficiency at the proposed optimal conditions. The mean values related to the influence of each factor at related levels might be calculated according to the assignment of the designed trials [38]. Thus, the influence of different levels of each examined factor on the size of resulted $KClO_4$ particles in the drying process was calculated and the results are given in Fig. 3. In fact, this figure presents the curves corresponding to the changes of the $KClO_4$ particle size due to the variation of the level of any studied factor.

The nature of non-solvent is a well-known factor which influences the size of resulted particles from the drying process. In this study, three different non-solvents, i.e., butyl acetate (BuAc), chloroform ($CHCl_3$), and dichloromethane (CH_2Cl_2), were utilized to precipitate the $KClO_4$ particles. The results in Fig. 3a exhibit that the nature of the non-solvent influences the size of micronized $KClO_4$ particles by drying process. Ideally, the target compound is insoluble completely in the resulted mixture of non-solvent/solvent during the spraying process, while the solvent is totally miscible with the non-solvent. Thus, spraying of the solution into the non-solvent results in a high supersaturated solution and, hence, the target solute nucleates. As a result, nature of the non-solvent could influence the morphology of the produced particles by its non-solvating power, interaction of non-solvent–solute, and its miscibility with the solvent.

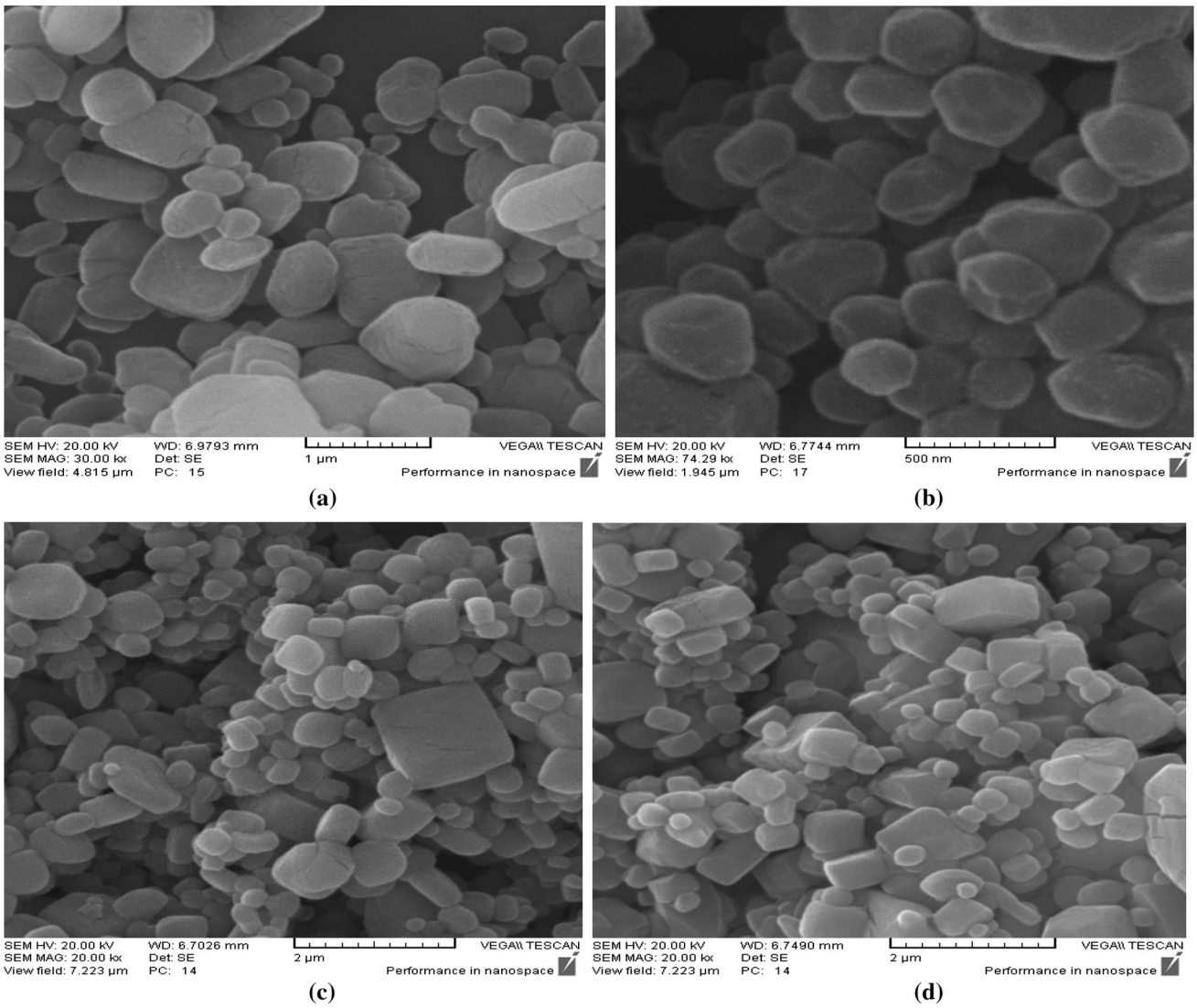
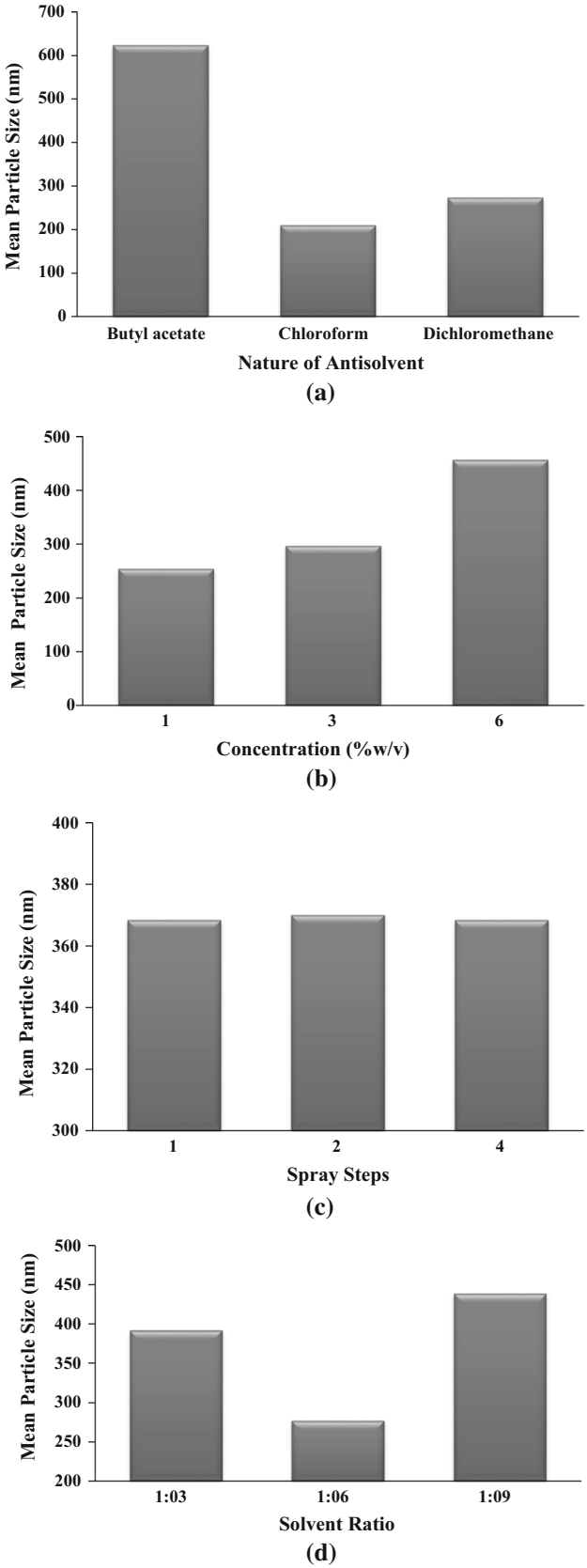


Fig. 2 SEM images for $KClO_4$ particles obtained by different runs of OA_9 (Table 1) via solvent/anti-solvent process: **a** run 5, **b** run 6, **c** run 7 and **d** run 8

The concentration of $KClO_4$ was also examined to identify its role on the size of resulted $KClO_4$ particles. As shown in Fig. 3b, concentration of $KClO_4$ effects on the particle size and among the studied levels of this factor (1, 3, and 6 % w/v), 3 % yields the best efficiency and fine product particles. Spray steps were investigated to determine their role on the particle size of resulted $KClO_4$. Figure 3c gives the investigated levels of this factor and the average results for them. As shown in this figure, variation in the spraying steps leads to the mild changes in the size of $KClO_4$ particles. Also, the effects of solvent to non-solvent (at three different ratios of 1:3, 1:6, and 1:9) on the particle size were examined. As shown in Fig. 3d, this factor has a considerable effect on the particle size of micronized $KClO_4$. Among the ratios studied in this investigation, 1:6 provided best efficiency for the micronization of potassium perchlorate.

The ANOVA results for the $KClO_4$ micronization experiments are presented in Table 2. As shown in this table, except spray steps, all tested factors (at 90 % confidence level) have a significant role in defining the particle size of $KClO_4$ resulted by spraying with non-solvent process. In other words, ANOVA results confirm the insignificant role of spray steps in controlling the size of $KClO_4$ particles micronized by spraying in non-solvent method.



◀**Fig. 3** Average particle size of $KClO_4$ obtained via solvent/anti-solvent process corresponds to the effect of each level for various factors: **a** nature of anti-solvent; **b** concentration of $KClO_4$; **c** spray steps; **d** solvent ratio

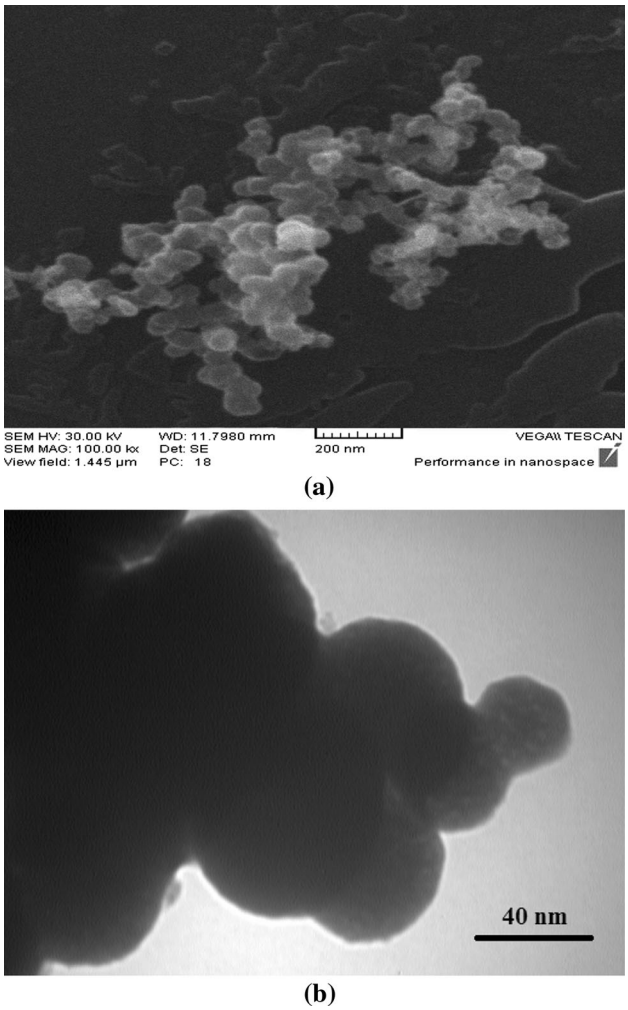


Fig. 4 **a** SEM image and **b** TEM image of prepared $KClO_4$ under optimum condition via solvent/anti-solvent process

3.2 Determination of optimal conditions of spraying in non-solvent process and preparation of $KClO_4$ nanoparticles

According to the ANOVA results and considering the average role of examining factors (Fig. 3), the optimal conditions for micronizing of $KClO_4$ by spraying in non-solvent procedure are as follows: 3 % $KClO_4$ concentration, $CHCl_3$ as non-solvent, and solvent to non-solvent ratio of 1:6. Meanwhile, the size of $KClO_4$ particles at these optimum

Table 3 The operating conditions of the SAS process in nanoparticles production

Flow rate of CO_2 (mL/min)	Solution flow rate (mL/min)	Concentration (% w/v)	Temperature (°C)	Pressure (bar)	Solvent
50	3	2	80	50	DMF

conditions of the spraying in non-solvent procedure might be predicted via the following expression [38, 39]:

$$Y_{\text{opt}} = \frac{T}{N} + \left(A - \frac{T}{N}\right) + \left(C - \frac{T}{N}\right) + \left(R - \frac{T}{N}\right)$$

where T/N is average size of resulted $KClO_4$ particles from all of the trials; however, T is the overall size of $KClO_4$ particle resulted from all runs of Table 1, N is the total number of performed trials, Y_{opt} is the size of $KClO_4$ particles at optimal conditions, A, C, and R, respectively, are the calculated average size of $KClO_4$ particles contributed to the non-solvent identify, $KClO_4$ concentration, and solvent to non-solvent ratio at their optimal level (minimum particle size for each factor resulted from the average effect of each factor as shown in Fig. 3). The equation was utilized for the estimation of the particle size of $KClO_4$ at optimal conditions (at 90 % confidence level) and revealed an average particle size of about 46 ± 5 nm.

The size of formed particles by spraying in non-solvent process varies inversely with the local supersaturating resulted in the intermediate of solution droplets and non-solvent media during the solution spraying period and dissolving of the droplets in the non-solvent. Meanwhile, after creating the initial $KClO_4$ nuclei, the process proceeds by the competition between further nucleation and the growth of the existing nuclei [32]. Therefore, for the formation of $KClO_4$ nanoparticles during the spraying in non-solvent process, the rate of nucleation should be higher than the particles growth to achieve a large number of ultrafine particles. In other words, Taguchi robust design permits optimization of the spraying in non-solvent variables process experimentally and determination of the required conditions for predominating of nucleation process on the particle growth which leads to the formation of $KClO_4$ nanoparticles. On the other hand, $KClO_4$ nanoparticles were prepared at the proposed optimal conditions of the spraying in non-solvent process. Figure 4a gives the SEM image of resulted $KClO_4$ nanoparticles. It was found that the average size of $KClO_4$ particles obtained at these optimal conditions is about 41 nm which is in agreement with the predicted range. This result is confirmed by TEM image of $KClO_4$ nanoparticles (Fig. 4b).

3.3 Preparation of $KClO_4$ nanoparticles by supercritical carbon dioxide anti-solvent (SAS) process

$KClO_4$ nanoparticles were also fabricated by supercritical carbon dioxide anti-solvent process under the operating conditions presented in Table 3. These conditions for the SAS were selected based on the preliminary tests and the previous studies on the energetic materials micronization by supercritical carbon dioxide anti-solvent process [15, 35]. Figure 5 shows the SEM image of the resulted $KClO_4$ nanoparticles by SAS procedure. As shown in this figure, the average size of $KClO_4$ particles obtained by the SAS process is about 55 nm.

3.4 Characterization of $KClO_4$ nanoparticles by XRD

Figure 6 shows the XRD patterns of the potassium perchlorate nanoparticles obtained at optimal conditions of the spraying in non-solvent route and SAS process. As shown, all the diffraction peaks in the figures could be indexed to be in agreement with the orthorhombic structure of $KClO_4$ from PDF Card No. 01-076-1852. These patterns indicated that the resulted potassium perchlorate nanoparticles by both routes have a similar crystalline structure with the high purity.

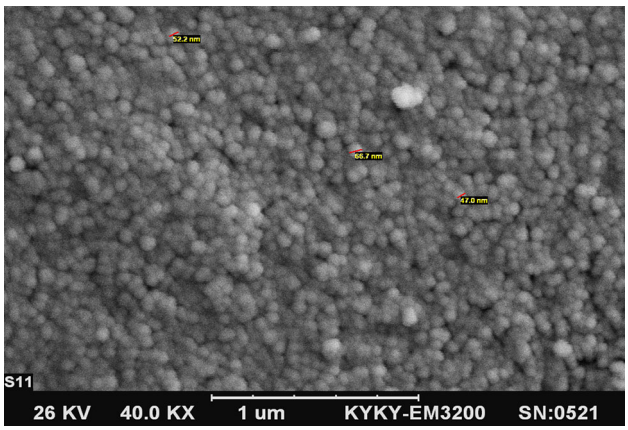


Fig. 5 SEM image of prepared $KClO_4$ via supercritical anti-solvent (SAS) process

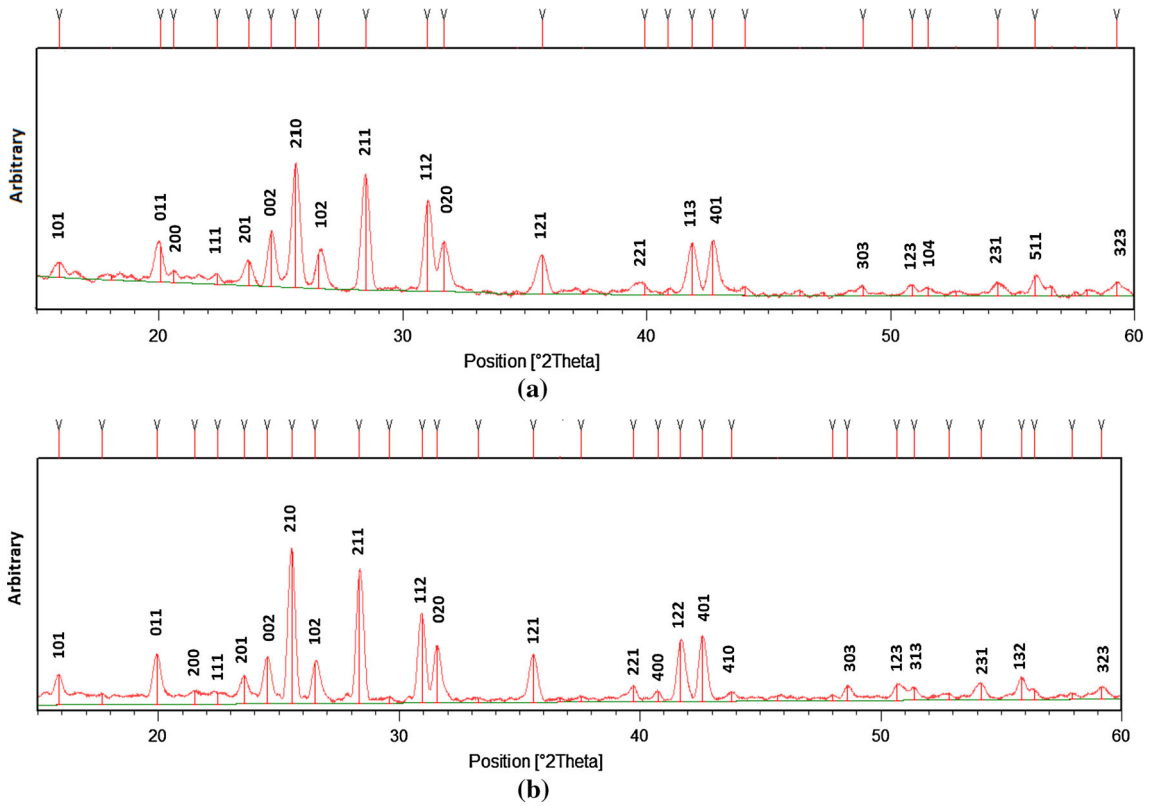


Fig. 6 XRD patterns of the prepared $KClO_4$ via **a** solvent/anti-solvent route and **b** supercritical anti-solvent (SAS) process

4 Conclusion

In this investigation, $KClO_4$ nanoparticles were successfully fabricated by two different processes, i.e., spraying in non-solvent process and supercritical carbon dioxide anti-solvent route. The Taguchi experimental design was utilized to optimize the operating conditions of the spraying in non-solvent process with the aim of obtaining $KClO_4$ nanoparticles. The results showed that spray steps have no significant effects on the size of $KClO_4$ particles resulting from spraying in non-solvent process, while other studied factors, i.e., $KClO_4$ concentration, nature of non-solvent, and solvent to non-solvent ratio, require tuning in order to efficiently control the product particle size. It was found that $KClO_4$ nanoparticles with average particle size of 41 nm might be prepared at optimal conditions of the spraying in non-solvent process. However, utilizing SAS process resulted in $KClO_4$ nanoparticles with the average size of about 55 nm.

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