Radune et al. International Journal of Mechanical and Materials Engineering (2022) 17:1
https://doi.org/10.1186/s40712-021-00140-8

International Journal of Mechanical and Materials Engineering

ORIGINAL PAPER Open Access

Use of Taguchi method for high energy ball milling of $CaCO_3$

Maya Radune[1*], Svetlana Lugovskoy[2], Yaniv Knop[1] and Avigdor Yankelevitch[2]

Abstract

Taguchi's method was applied to investigate the effect of main high energy ball milling (HEBM) parameters: milling time (MT), ball to powder weight ratio (BPWR), and milling speed (MS) on the $CaCO_3$ crystallite size. The settings of HEBM parameters were determined by using the *L9* (3^3) orthogonal experiments array (OA). The as-received and milled powders were characterized by X-ray diffraction (XRD) scanning electron microscopy (SEM), Fourier transform infrared (FTIR) spectroscopy. The crystallite size of $CaCO_3$ varied between 140 and 540 nm depending on the HEBM conditions. The analysis of variance (ANOVA) was used to find the significance and percentage of contribution of each milling parameter. It was established that the MT is the most effective parameter followed by MS and BPWR. A confirmation test was carried out with a 90% confidence level to illustrate the effectiveness of the Taguchi optimization method. The optimum milling parameter combination was determined by using the analysis of signal-to-noise (S/N) ratio. Based on the S/N ratio analysis, optimal HEBM conditions were found MT 10 h, MS 600 revolutions per minute (rpm), BPWR 50:1.

Keywords: Taguchi's technique, ANOVA, High energy ball milling, $CaCO_3$

Introduction

Calcium carbonate ($CaCO_3$) has been widely studied due to its chemical stability and mechanical reinforcement ability (Chen et al. 2010 and Tanniru et al. 2005). $CaCO_3$ is one of the cheapest commercially available inorganic materials (Kumar et al. 2014), and therefore has innumerable industrial applications: it is used in paints, inks, coatings, paper products, plastics, and films (Garcia et al., 2002). Research has focused on the effects of minimizing crystallite size of $CaCO_3$ on different properties (Krumpfer et al. 2013). Lately, crystallite size has taken on importance in tailoring final properties of $CaCO_3$ materials (Minkowicz et al. 2021, d'Amora et al. 2020, Safaei et al. 2021).

Small crystallite size $CaCO_3$ is usually produced by a wet chemical precipitation technique (Tsuzuki et al. 2000 Minkowicz et al. 2021, d'Amora et al. 2020), yielding precipitated $CaCO_3$ with a needle-like crystalline shape. However, the technique causes considerable agglomeration of particles during synthesis, requiring the precise control of a number of operation parameters, which determines the size, crystal structure, and morphology of the particles. In addition, precipitation techniques require numerous process steps while high-energy planetary ball milling is an efficient and simple method for the fabrication of sub-micron or nanostructured powder and leads to a better distribution of crystallite and particles size (Suryanarayana et al. 2001). Since precipitation techniques need numerous process steps, HEBM is an efficient and simple method for the fabrication of sub-micron or nanostructured powder materials (Suryanarayana et al. 2001). Several investigators have explored the mechanism and limit of the grain size refinements achievable during ball milling in materials with different crystal structures (Suryanarayana et al. 2001, Magini et al. 1996, and Koch et al. 1996). HEBM is a process involving a number of both independent and interdependent variables. In the planetary ball milling, the main factors that affect particle size reduction

* Correspondence: radune@ariel.ac.il
[1]Civil Engineering Department, Ariel University, Kiryat Hamada, 40700 Ariel, Israel
Full list of author information is available at the end of the article

include MS, size of balls, BPWR, medium of milling, MT, etc. (Davis et al. 1987).

The Taguchi method provides a simple, efficient, and systematic approach to determine optimal parameters (Gopalsamy et al. 2009). Compared to the factorial method, instead of testing all possible combinations of parameters available, the Taguchi method provides a more simplified way to set up the combination of experimental parameters (Ngo et al. 2018). In recent years, statistical experimental designs have been employed to determine optimal HEBM parameters (Radune et al., 2015). This study aimed to determine the effect of HEBM parameters (MS, MT, and BPWR) on crystallite sizes of $CaCO_3$ powder by applying the Taguchi method.

The above parameters were optimized according to the calculated S/N ratio of parameters. Moreover, an ANOVA is employed to determine statistically significant parameters. Each obtained product was characterized using XRD, STEM, and FTIR spectroscopy.

Materials and methods

$CaCO_3$ micron size powder, supplied by Adacal Mineraller LTD, was used as starting material. The morphology of this powder is shown in Fig. 1. The raw $CaCO_3$ particles are micron size and irregular in shape. $CaCO_3$ powder underwent HEBM in a planetary ball mill (Retsch PM 100, Germany) using container and balls (10 mm diameter) made from chromium hardened steel. The phase compositions of the initial and milled powders were determined by XRD using a Panalytical X'Pert Pro X-ray Diffractometer with CuK_α radiation (λ = 0.154 nm), operating at 40 kV and 40 mA. Data collection was performed by step scanning of the specimen over the 2θ : 20–70° angular range in steps of 0.05° with 3 s per step. The XRD line profile parameters treated according to the Rietveld procedure using PANalytical X'Pert HighScore Plus v3.0e software. The crystallite sizes of the milled powders were determined from a broadening of XRD peaks by the Williamson-Hall (WH) method (Williamson et al. 1953). The morphology of the initial and milling powders was examined by using STEM (MAIA3 TESCAN) used in SEM mode. Thus, the abbreviation SEM will be used further on. The FTIR spectrum curves of the $CaCO_3$ milling powders were obtained over the wavelength of 4000–450 cm^{-1} by using Thermo-Scientific Nicolet IS10 in standard KBr-based tablets formed under pressure of 3 atm.

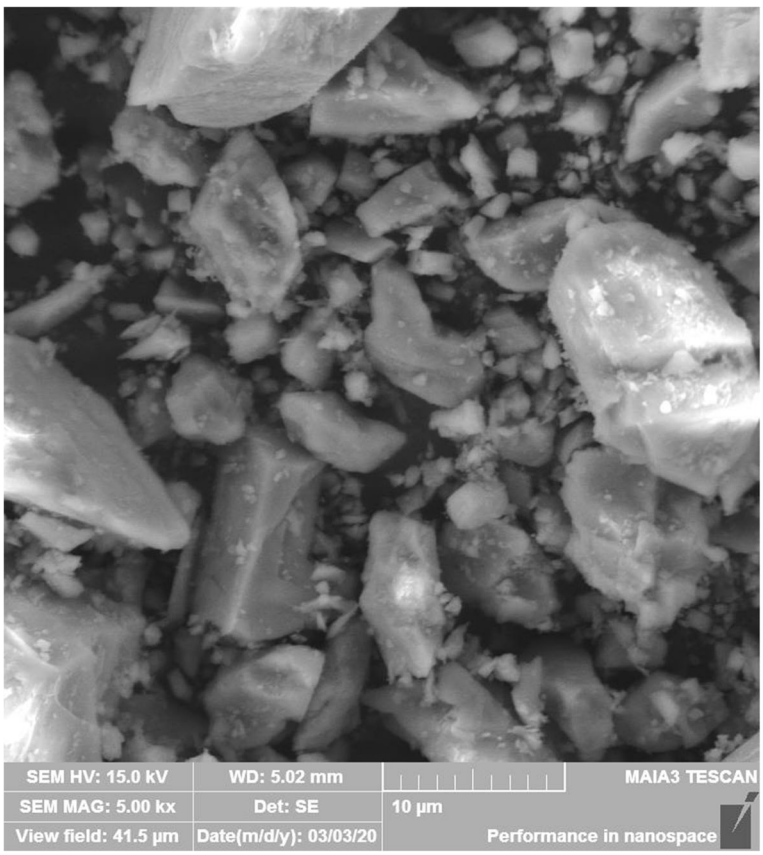

Fig. 1 SEM images of starting $CaCO_3$ powder

Table 1 Process parameters and levels

Levels Parameters	1	2	3
MT, [h]	5	10	30
MS, [rpm]	200	400	600
BPWR	10	20	50

Experimental design

Taguchi's parameter design approach is applied for optimization of the HEBM process variable of $CaCO_3$. Many parameters are used in the ball milling process. However, the parameters that have been tested most for optimization are the MS, MT, and BPWR (Rud et al. 2012). This indicates that these three parameters play an important role in determining the effectiveness of the milling. There is no conclusive evidence on the best values of the above parameters in previous works (Suryanarayana, 2001).

The influences of MS (200, 400, and 600 rpm), MT (5, 10, and 30 h) and BPWR (10:1, 20:1, and 50:1 on the crystallite size of $CaCO_3$ powder were investigated (Table 1).

Numbers 1, 2, and 3 represent the lowest, mid, and highest levels, respectively.

The minimum number of experiments that are required to conduct the Taguchi method can be calculated based on the degrees of freedom approach (Eq. 1)

$$DOF = (P-1)F + (P-1)Q + 1 \quad (1)$$

where DOF is the desired degree of freedom, F is number of independent (or) input variables, P is their chosen levels, Q is number of interactions needed in the study and 1 is the average. Taguchi's methodology was applied for the three factors selected without considering the interaction effect between them, and, therefore, the total number of DOF is 1+3 (3–1) = 7. The number of experimental trials must be equal to or larger than the DOF for performing experiments in process optimization. The most appropriate OA in this case is *L9* (3^3) (Table 2).

If the interaction effect is considered in the experiment, a higher orthogonal array must be selected. The small size of the experiments and the fact that they seem to provide satisfactory results are the two reasons that orthogonal arrays are preferred for experimental designs. The verification of results comes from running experiments by carrying out a confirmation test at the predicted optimal conditions.

Nine trial runs with certain factor level combinations determined from the array were carried out randomly and repeatedly. Qualitek-4 software was used to assist in selecting the runs in the experimental design in random order. The experiments were replicated three times for statistical purposes. Thus, 9 × 3 = 27 are required. This is far less than the 3 × (3^3) = 81 experiments that would be needed according to full factorial design.

All calculations were analyzed using Qualitek-4 software.

Results and discussions

The FTIR spectra of the raw material and typical milling $CaCO_3$ powders are shown in Fig. 2. A sharp peak at 3642 cm^{-1} was observed in the FTIR spectrum due to the presence of the stretching mode of OH^- in the raw material (Feng et al. 2016). The absorption peaks of CO_3^{2-} appeared around 710–719, 860–871, and 1400–1410 cm^{-1}. The presence of these bands confirmed that each milled powder was in the form of $CaCO_3$ (Feng et al. 2016). These findings were in good agreement with the XRD examination, i.e., that $CaCO_3$ did not decompose during HEBM.

Each milling powder was identified as calcite or aragonite (Fig. 3). No impurities were observed during the XRD examination. The diffraction Bragg peaks are broadened and reduced in intensity, which can be

Table 2 The experimental layout of the L9 (3^3) OA

Experiment number	Parameters and trial conditions			Responses (raw data)		
	MT	MS	BPWR	R_1	R_2	R_3
1	1	1	1	$Y_{1,1}$	$Y_{1,2}$	$Y_{1,3}$
2	1	2	2	$Y_{2,1}$	$Y_{2,2}$	$Y_{2,3}$
3	1	3	3	$Y_{3,1}$	$Y_{3,2}$	$Y_{3,3}$
4	2	1	2	$Y_{4,1}$	$Y_{4,2}$	$Y_{4,3}$
5	2	2	3	$Y_{5,1}$	$Y_{5,2}$	$Y_{5,3}$
6	2	3	1	$Y_{6,1}$	$Y_{6,2}$	$Y_{6,3}$
7	3	1	3	$Y_{7,1}$	$Y_{7,2}$	$Y_{7,3}$
8	3	2	1	$Y_{8,1}$	$Y_{8,2}$	$Y_{8,3}$
9	3	3	2	$Y_{9,1}$	$Y_{9,2}$	$Y_{9,3}$

R_1, R_2, and R_3 are response values for three replicates of each trial. The 1s, 2s, and 3s denote the levels 1, 2, and 3 of the parameters. Y_{ij} are $CaCO_3$ crystallite sizes

Table 3 The $CaCO_3$ crystallite size and S/N ratios (average ± SD, replicated 3 times)

Experiment number	Average crystallite size, nm	*S/N* ratio, dB
1	504 ± 51	− 54.05
2	300 ± 46	− 49.58
3	185 ± 25	− 45.33
4	215 ± 13	− 46.64
5	139 ± 36	− 42.84
6	199 ± 27	− 45.97
7	287 ± 31	− 49.41
8	260 ± 47	− 48.30
9	230 ± 35	− 47.23

related to the crystallite size reduction and the internal strain in the milling powder. WH analysis showed that the strain of the milled powders is above 0; therefore, the broadened peaks indicate crystallite size only. In Table 3, the average $CaCO_3$ crystallite size, obtained in three repeated experiments for each of nine trial conditions, are presented. As compare to the average data the measured crystallite sizes are in the range of 136–538 nm. Each sample has its own conditions (different BPWR, MS and MT) which causes the changes in the XRD peaks. Note that each of the selected parameters affects the changes in peaks in its own power, which was analyzed employing the Taguchi procedure.

The SEM images in Fig. 4 show that the milling conditions did not strongly affect the morphological property of each product and that a similar crystal morphology was observed.

Statistical optimization

The value of signal-to-noise ratio (S/N) was used to determine the optimal and most influential parameters to the crystallite size of $CaCO_3$ HEBM powder. The S/N ratio formula for the static design was divided into three categories: 'nominal the best,' 'larger the better,' and 'smaller the better' (Phadke, 1989). A "smaller the better" formula (Eq. 2) was chosen for the analysis of the experimental results because lower crystallite size was desirable:

$$S/N = -10\ \log\left[\frac{1}{n}\sum_{i=1}^{n} y_i^2\right] \tag{2}$$

where n is the number of experiments in the OA, and y_i is the ith measured value.

Data experiment results and the computed S/N ratio are presented in Table 3.

The differences between obtained values were strongly dependent on the milling conditions.

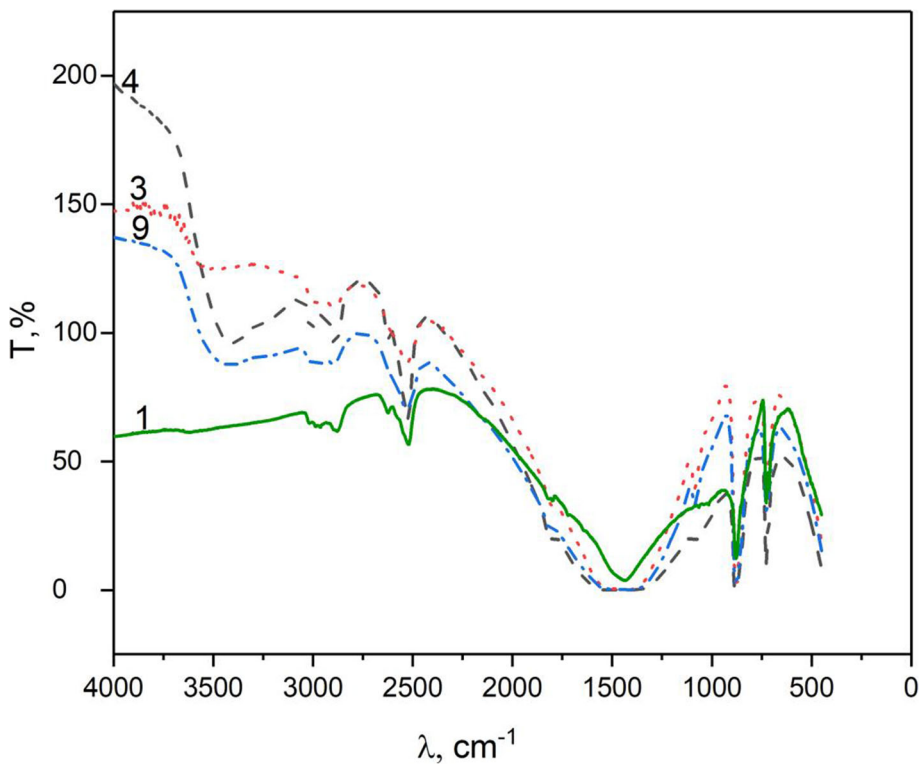

Fig. 2 FTIR spectra curves for non-milled and milled $CaCO_3$ powders. The number 1 indicates the non-milled powder. The numbers 3, 4, and 9 indicate the experiment number

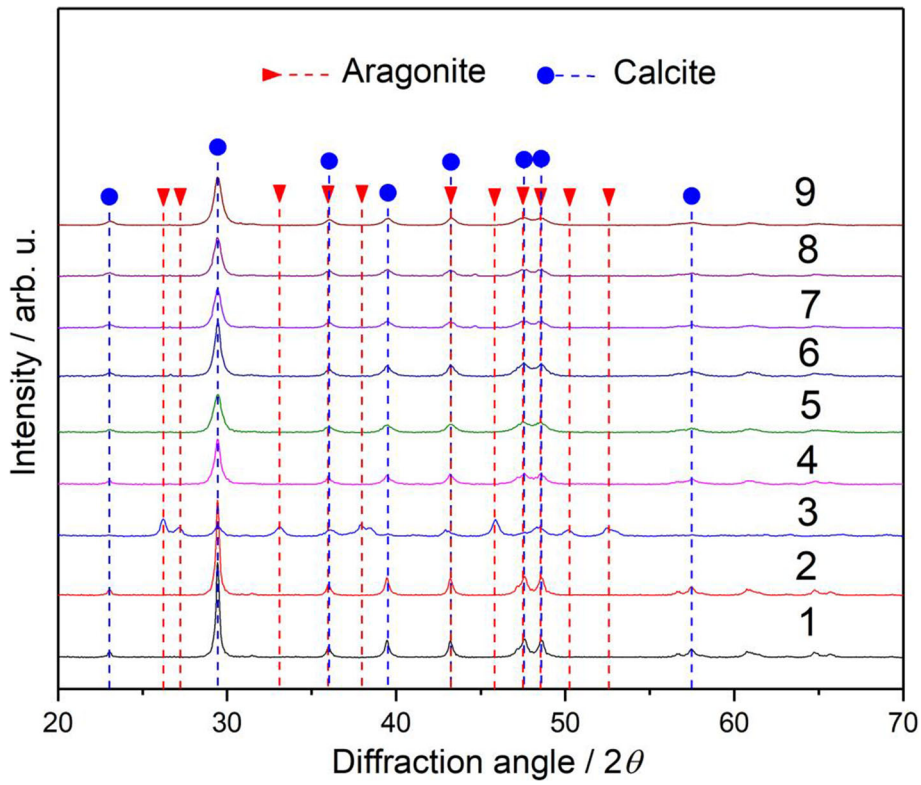

Fig. 3 XRD patterns for the HEBM $CaCO_3$ powders. The numbers from 1 to 9 indicate the experiment number

Since the experimental design is orthogonal, it is possible to separate the effect of each parameter at different levels (Ross, 1996). The mean S/N ratio is the average of the S/N ratio for each parameter at different levels (Taguchi et al. 2005). For example, the effect of MS at level 1 (MS_1) (experiments 1, 4, and 7) is calculated as follows:

$$MS_1 = \frac{1}{3}\left(\frac{S}{N_1} + \frac{S}{N_4} + \frac{S}{N_7}\right) \quad (3)$$

Thedifference between maximum and minimum Table 4.

The difference between maximum and minimum S/N ratios determines the main effect of the parameter. With greater delta (Δ) values for a parameter, the effect of the parameter on the process will correspond to a smaller variance of the output and generate better performance of the experiment (Külekcı, 2013). From the point of the "smaller the better" quality characteristic, the MT (Δ = 4.50) has the largest effect on the $CaCO_3$ crystallite size, while BPWR has the smallest effect (Δ = 3.58).

A parameter level corresponding to the maximum average S/N ratio is called the optimal level for that parameter (Thakur et al. 2012). Based on the S/N ratio analysis, the optimal conditions for smaller $CaCO_3$ crystallite size are MS_3 (600 rpm), MT_2 (10 h), and $BPWR_2$ (30:1) (Fig. 5).

Table 4 Response table for $CaCO_3$ crystallite size

Level	MT	MS	BPWR
1	− 49.65	− 50.03	− 49.44
2	− 45.15*	− 46.91	− 47.82
3	− 48.32	− 46.18*	− 45.86*
Delta Δ	4.50	3.85	3.58
Rank	1	2	3

*Optimal parameter level

Statistical analysis of variance (ANOVA)

The Taguchi method cannot judge and determine the effect of individual parameters on the entire process while the percentage contribution of individual parameters can be determined using analysis of variance ANOVA (Roy, 2001). Qualitek-4 software of the ANOVA module was employed to investigate the effect of process parameters. ANOVA results are presented in standard ANOVA tables (Tables 5 and 6).

From the results of ANOVA for $CaCO_3$ crystallite size, the last column in Table 5 illustrated the percentage contribution of each parameter. Percentage contribution is defined as the significant rate of the process parameters and larger values represent a more significant effect on the crystallite size of HEBM $CaCO_3$. Thus, the MT, MS

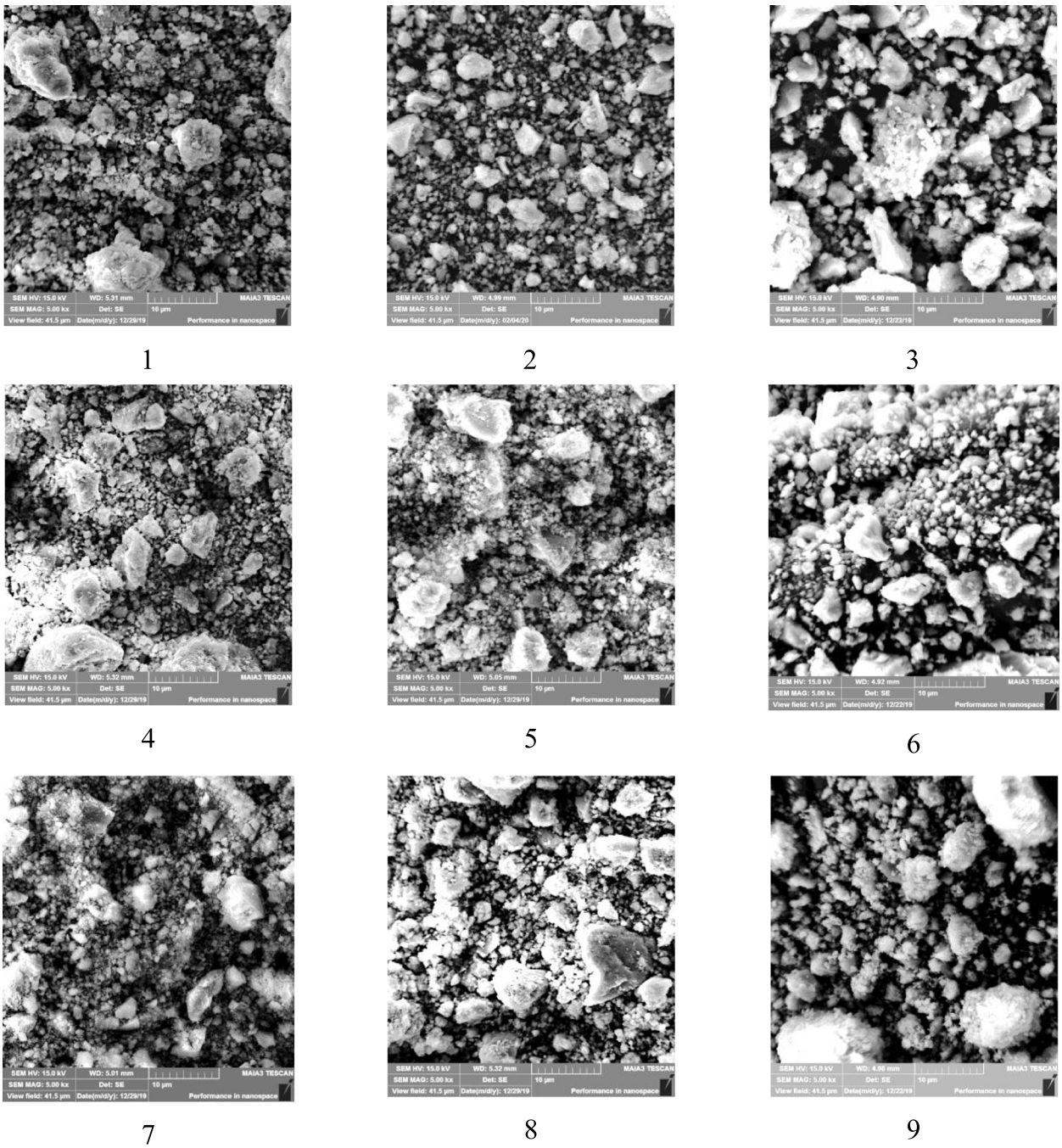

Fig. 4 SEM images of HEBM $CaCO_3$ powders. The numbers from 1 to 9 indicate the experiment number

and BPWR were found to be statistically significant at a confidence level of 90% (α = 0.1), with a contribution of 32.46%, 27.95%, and 21.09%, respectively. The S/N ratio exhibits a similar trend and is tabulated in Table 6. The ANOVA results closely match with the Taguchi ones.

Prediction of crystallite size under optimal conditions

The crystallite size is predicted at the optimal levels of milling parameters: MS_3, MT_2, and $BPWR_2$ (Eq.4).

$$u_0 = y_m + \sum_{i=1}^{n} \left(y_j - y_m \right) \tag{4}$$

where y_m is the total mean of the crystallite size y, n is the number of main milling parameters which significantly affect performance, and y_j is the mean measured values y for jth milling parameters corresponding to

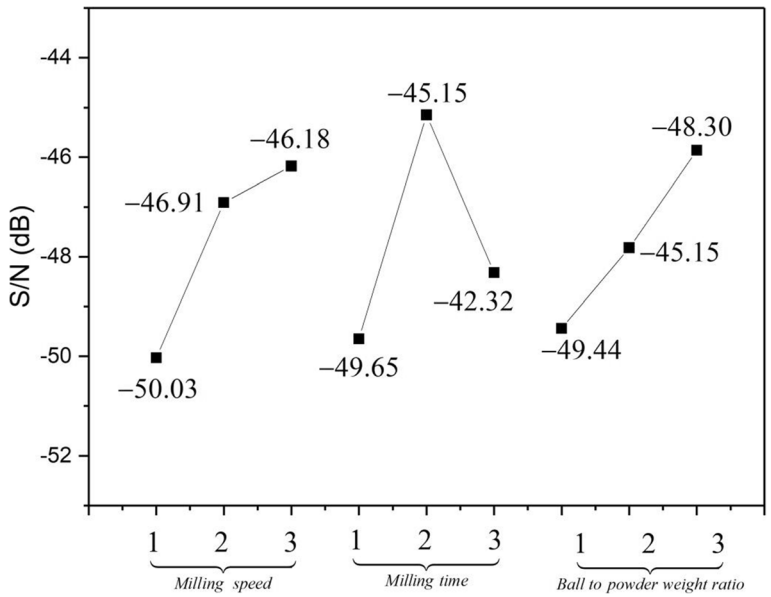

Fig. 5 Main effect of process parameters on S/N

optimal parameter level. According to these values, the crystallite size was computed as 77.03 nm.

The confidence interval (CI) was employed to verify the quality characteristics of the confirmation experiments. The CI for the predicted optimal values is calculated as follows (Eq. 5):

$$C.I. = \sqrt{F_\alpha(1, f_e) V_e \left[\frac{1}{n_{eff}} + \frac{1}{R}\right]} \tag{5}$$

where

$$n_{eff} = \frac{N}{1 + [DOF\ \ associated\ \ in\ \ the\ \ estimate\ \ of\ \ mean\ \ responce]} \tag{6}$$

$F_\alpha\ (1, f_e)$ is the F-ratio at 90% confidence level, f_e is the degree of freedom for error, V_e is variance of the error term (from ANOVA Table 4), R is sample size for confirmation experiments (R = 3), n_{eff} is number of effective measured results, and N is the total number of results or number of S/N ratios. The CI at 90% confidence level is calculated to be ± 59.48. Thus, with 90% confidence level, the estimated optimal $CaCO_3$ crystallite size is (77.03 ± 59.48)nm—i.e., the confirmation result should be within 17.55 and 136.51 nm.

Confirmation test

With the Taguchi optimization methodology, a confirmation test is required to validate the optimized condition. Three confirmation runs were conducted under optimal conditions (MS_3, MT_2 and $BPWR_2$). The average $CaCO_3$ crystallite size was found to be 115 nm. This result is within 90% confidence interval of predicted optimal $CaCO_3$ crystallite size value (122.69 nm). Therefore, confirmation tests depicted the successful optimization.

Conclusions

The Taguchi method of experimental design has been applied to obtain optimal process parameters for $CaCO_3$ powder using the HEBM process and was analyzed with the Taguchi L9 orthogonal array. The optimal results can be obtained by selecting the second level of the MT (10 h) and third level of the MS (600 rpm) and BPWR (50:1). It was concluded that MT is the most influential parameter followed by the MS and BPWR. Based on the ANOVA table, the percentage contributions of the MS,

Table 5 ANOVA table for $CaCO_3$ crystallite size

Factor	DOF (f)	Sum of squares (S)	Variance (V)	F-ratio (F)	Pure sum (S)	Contribution, P(%)
MS	2	95787.16	47893.58	23.82	91765.86	32.46
MT	2	83014.33	41507.16	20.64	78993.03	27.95
BPWR	2	63647.94	31823.97	15.83	59626.64	21.09
Other error	20	40213.00	2010.65			18.50
Total	26	282662.43				100%

Table 6 ANOVA table for S/N ratio

Factor	DOF (f)	Sum of squares (S)	Variance (*V*)	F-ratio (F)	Pure sum (*S*)	Contribution, *P*(%)
MT	2	32.52	16.26	7.69	28.29	35.07
MS	2	24.34	12.17	5.75	20.11	24.93
BPWR	2	19.58	9.79	4.63	15.35	19.03
Other error	2	4.23	2.11			20.97
Total	8	80.68				100%

MT, and BPWR are 32.46%, 27.95%, and 21.09%, respectively at 90% confidence level. In addition, the confirmation tests using the predicted optimal parameters observed that the average $CaCO_3$ crystallite size is within the expected range of the values for confidence limit, which makes the results trustworthy.

Abbreviations

HEBM: High energy ball milling; MT: Milling time; BPWR: Ball to powder weight ratio; MS: Milling speed; OA: Orthogonal experiments array; XRD: X-ray diffraction; STEM: Scanning transmission electron microscopy; SEM: Scanning electron microscopy; FTIR: Fourier transform infrared spectroscopy; DOF: Desired degree of freedom; S/N: Signal-to-noise ratio; ANOVA: Analysis of variance; WH: Williamson-Hall; CI: Confidence interval

Acknowledgements

The authors thank Dr. A. Kossenko and N. Litvak for assistance with XRD and STEM.

Authors' contributions

MR and SL set up the experiment model. AY performed HEBM experiments. MR and SL used Qualitek-4 software to set up the Taguchi experimental design, to calculate and to analyze the results. YK helped project administration and resources. YK supervised the study. All authors read and approved the final manuscript.

Funding

This research received no external funding.

Availability of data and materials

All data generated or analyzed during this study are included in this published article.

Declarations

The authors declare that on acceptance of the manuscripts for publication the data used for the work will be available to all concerned. It will be interesting for both scientific and industrial purpose especially to all $CaCO_3$ industries.

Competing interests

The authors declare that they have no competing interests.

Author details

[1]Civil Engineering Department, Ariel University, Kiryat Hamada, 40700 Ariel, Israel. [2]Chemical Engineering Department, Ariel University, Kiryat Hamada, 40700 Ariel, Israel.

Received: 12 January 2021 Accepted: 7 December 2021
Published online: 04 February 2022

References

Chen, X., Zhu, Y., Zhou, B., Guo, Y., Gao, W., Ma, Y., … Wang, Z. (2010). Hydrophilic CaCO3 nanoparticles designed for poly(ethylene terephthalate). *Powder Technol*, *204*(1), 21–26. https://doi.org/10.1016/j.powtec.2010.07.002.

Davis, R. M., & Koch, C. C. (1987). Mechanical alloying of brittle components: silicon and germanium. *Scripta Metall*, *21*(3), 305–310. https://doi.org/10.1016/0036-9748(87)90218-3.

d'Amora, M., Liendob, F., Deorsolab, F. A., Bensaidb, S., & Giordania, S. (2020). Toxicological profile of calcium carbonate nanoparticles for industrial applications. *Colloids Surf B: Biointerfaces*, *190*, 110947. https://doi.org/10.1016/j.colsurfb.2020.110947.

Feng, J., Wu, G., & Qing, C. (2016). Biomimetic synthesis of hollow calcium carbonate with the existence of the agar matrix and bovine serum albumin. *Mater Sci Eng C Mater Biol Appl*, *58*, 409–411. https://doi.org/10.1016/j.msec.2015.09.005.

Garcia, F., Le Bolay, N., & Frances, C. (2002). Changes of surface and volume properties of calcite during a batch wet grinding process. *Chem Eng J*, *85*(2-3), 177–187. https://doi.org/10.1016/S1385-8947(01)00152-8.

Gill A. S, Thakur A & Kumar S (2012) Effect of deep cryogenic treatment on the surface roughness of OHNS die steel after WEDM. Int J Appl Eng Res 7:1508-1512.

Gopalsamy, B. M., Mondal, B., & Ghosh, S. (2009). Taguchi method and anova: an approach for process parameters optimization of hard machining while machining hardened steel. *J Sci Ind Res*, *68*, 689–695.

Koch, C. C. (1996). Amorphization of single composition powders by mechanical milling. *Scripta Materialia*, *34*(1), 21–27. https://doi.org/10.1016/1359-6462(95)00466-1.

Krumpfer, J. W., Schuster, T., Klapper, M., & Müllen, K. (2013). Make it nano-Keep it nano. *Nano Today*, *8*(4), 417–438. https://doi.org/10.1016/j.nantod.2013.07.006.

Külekcı, M. K. (2013). Analysis of process parameters for a surface-grinding process based on the Taguchi method. *Mater Tehnol*, *47*, 105–109.

Kumar, V., Dev, A., & Gupta, A. P. (2014). Studies of poly(lactic acid) based calcium carbonate nanocomposites. *Composites*, *56*, 184–188. https://doi.org/10.1016/j.compositesb.2013.08.021.

Magini, M., Iasonna, A., & Padella, F. (1996). Ball milling: an experimental support to the energy transfer evaluated by the collision model. *Scripta Materialia*, *34*(1), 13–19. https://doi.org/10.1016/1359-6462(95)00465-3.

Minkowicz, L., Dagan, A., Uvarov, V., & Benny, O. (2021). Controlling calcium carbonate particle morphology, size, and molecular order using silicate. *Materials*, *14*(13), 3525–3535. https://doi.org/10.3390/ma14133525.

Ngo, M. T., Hoang, V., & Hoang, S. V. (2018). Taguchi-fuzzy multi-response optimization in fly cutting process and applying in the actual hobbing process. *Int J Mech Mater Eng*. https://doi.org/10.1186/s40712-018-0092-z.

Phadke, M. S. (1989). Quality Engineering using Design of Experiments. In *Quality engineering using robust design*. New Jersey: Englewood Cliffs.

Radune, M., Zinigrad, M., & Frage, N. (2015). Optimization of high energy ball milling parameters for synthesis of TixAl1-xN powder. *J Nano Res*, *38*, 107–113. https://doi.org/10.4028/www.scientific.net/JNanoR.38.107.

Ross, P. J. (1996). *Taguchi techniques for quality engineering. s.l.* Mcgraw-Hill International Editions.

Roy, R. K. (2001). *Design of experiments using the taguchi approach*, (s.l. ed.,). A Wiley-Interscience Publication.

Rud, A. D., & Lakhnik, A. M. (2012). Effectof carbon allotropes on the structure and hydrogen sorption during reactive ball-milling of Mg-C powder mixtures. *Int J Hydrogen Energy*, *37*(5), 4179–4187. https://doi.org/10.1016/j.ijhydene.2011.11.123.

Safaei B, Davodian E, Fattahi A.M., Fattahi A.M., Asmael M. (2021) Calcium carbonate nanoparticles effects on cement plast properties. Microsyst Technol 27: 3059–3076. https://doi.org/10.1007/s00542-020-05136-6, 8

Suryanarayana, C. (2001). Mechanical alloying and milling. *Prog. Mater. Sci*, *46*(1-2), 1–184. https://doi.org/10.1016/S0079-6425(99)00010-9.

Suryanarayana C, Ivanov E., Boldyrev V. V (2001) The science and technology of mechanical alloying Materi. Sci Eng A 304: 151-158.

Taguchi, G., Chowdhury, S., & Wu, Y. (2005). *Taguchi's Quality Engineering Handbook*, (s.l ed.,). John Wiley&Sons.

Tanniru, M., & Misra, R. D. K. (2005). On enhanced impact strength of calcium carbonate-reinforced high density polyethylene composites. *Materials Sci Eng A*, *405*(1-2), 178–193. https://doi.org/10.1016/j.msea.2005.05.083.

Tsuzuki, T., Pethick, K., & McCormick, P. G. (2000). Synthesis of $CaCO_3$ nanoparticles by mcchanochemical processing. *J Nanopart Res*, *2*(4), 375–380. https://doi.org/10.1023/A:1010051506232.

Williamson, G. K., & Hall, W. H. (1953). X-ray line broadening from filed aluminium and wolfram. *Acta Met*, *1*(1), 22–31. https://doi.org/10.1016/0001-6160(53)90006-6.

Publisher's Note