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Influence of Nano-Silica (SiO_2) Content on Mechanical Properties of Cement-Bonded Particleboard Manufactured from Lignocellulosic Materials

Utjecaj sadržaja nanočestica silicijeva dioksida (SiO_2) na mehanička svojstva cementne ploče iverice proizvedene od lignoceluloznih materijala

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ABSTRACT • *The influence of Nano-SiO_2 (NS) content and lignocellulosic material addition on hydration behavior of cement paste was studied through measurement of hydration temperature, initial and final setting time of cement paste and compressive strength of hardened cement paste. Besides, the amount of NS, particle size of reed and bagasse as lignocellulosic materials and bagasse to reed particles weight ratio were selected as manufacturing variables for cement-bonded particleboard (CBPB) each at five levels. The relationships between independent parameters and output variables (modulus of rupture (MOR), modulus of elasticity (MOE) and internal bonding (IB)) were modeled using response surface methodology (RSM) based on mathematical model equations (second-order multiple linear regression model) by computer simulation programming. The results indicated that cement pastes containing 3 wt.% Nano-SiO_2 content mixed with milled reed or bagasse particles enhanced maximum hydration temperature; however, the time of reaching the main rate peak shortened. Besides, the increase of SiO_2 replacement shortened the setting time. On the other hand, using reed particles, initial and final setting times of cement prolonged, while bagasse particles shortened initial and final setting times. Analysis of variance (ANOVA) was performed to determine the adequacy of the mathematical model and its respective variables. The interaction effect curves of the independent variables obtained from simulations showed a good agreement between the measured MOR, MOE and IB of CBPB and predicted values obtained by the developed models, and hence, the proposed concept was verified.*

Key words: *cement-bonded particleboard, Nano-Silica, reed, bagasse, hydration, RSM*

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SAŽETAK • *Utjecaj sadržaja nanočestica silicijeva dioksida (NS) i dodatka lignoceluloznih tvari ispitivan je na temelju hidratacijskog ponašanja cementne paste uz pomoć mjerenja temperature hidratacije, početnoga i završnog vremena vezanja cementne paste te tlačne čvrstoće očvrsnute cementne paste. Od varijabli koje utječu na svojstva cementnih ploča iverica (CBPB) ispitivana je količina NS-a, veličina čestica lignoceluloznih materijala (trske i otpadaka u preradi šećerne trske) te težinski omjer različitih lignoceluloznih materijala. Za svaku varijablu odabrano je pet vrijednosti. Odnos između nezavisnih parametara i izlaznih varijabli – modula loma (MOR), modula elastičnosti (MOE) i čvrstoće raslojavanja (IB) – modeliran je s pomoću metodologije odziva površine (RSM-a) i računalnim simulacijskim programiranjem utemeljen na jednadžbama matematičkih modela (model višestruke linearne regresije drugoga reda). Rezultati su pokazali da cementne paste koje (težinski) sadržavaju 3 % čestica NS-a pomiješanih s mljevenim česticama trske ili otpadaka u preradi šećerne trske pokazuju povećanje maksimalne temperature hidratacije, no skraćeno je vrijeme postizanja maksimuma. Osim toga, s povećanjem udjela NS-a skraćeno je vrijeme vezanja cementne paste. Nasuprot tome, primjenom čestica trske produljeno je početno i završno vrijeme vezanja cementa, a primjenom čestica od otpadaka u preradi šećerne trske skraćuje se početno i završno vrijeme vezanja cementa. Kako bi se utvrdila adekvatnost matematičkog modela i njegovih odgovarajućih varijabli, provedena je analiza varijance (ANOVA). Krivulje interakcije nezavisnih varijabli dobivenih iz simulacija pokazale su dobru podudarnost izmjerenih vrijednosti MOR-a, MOE-a i IB-a cementnih ploča iverica s pretpostavljenim vrijednostima dobivenim razvijenim modelima, te je stoga predloženi koncept potvrđen.*

Ključne riječi: *cementna iverica, nanočestice silicijeva dioksida, trska, ostatci pri preradi šećerne trske, hidratacija, RSM*

1 INTRODUCTION
1. UVOD

Cement-bonded particleboard is used widely in wall lining in public buildings, external cladding, protective elements for fireproofing, specialized flooring, etc. It is a wood-based composite manufactured from wood-based materials or other lignocellulosic materials and a mineral binder under high pressure. Large quantities of lignocellulosic-based material are produced every year in the world. These materials are investigated to produce cement-bonded particleboards. In the last decade, research has beeb carried out on a wide range of annual plants species and agricultural residues including reed stalk (Alpar *et al.,* 2012), wheat straw (Soroushian *et al.,* 2004), coconut (Olorunnisola, 2009; Almeida *et al.,* 2002), bagasse (Aggarwal, 1995; Nazerian and Hosiny Eghbal, 2013), oil palm (Hermawan *et al.,* 2001), flax (Aamr-Daya *et al.,* 2008), rice husk (Ciannamea *et al.,* 2010), bamboo (Das *et al.,* 2012), corn (Jarabo *et al.,* 2013), groundnut hulls (Ayobami *et al.,* 2013), vine stalk (Rangavar *et al.,* 2014), tea (Sapuan *et al.,* 2011) etc.

All of these materials, such as giant reed (*Arundo donax L.*), can be applied for producing different types of composite materials. However, these materials have some negative effects on the properties of composites. Due to a rather high concentration of extractives compared to wood materials, higher inhibitory effects of available extractives in non-wood materials or agricultural residues can be expected on the hydration process of cement paste. Presence of these compounds increases the proportion of unhydrated cement particles and decreases the strength of the cement-bonded particleboard (Wei *et al.,* 2003). Different treatments can be used to minimize the effects of the inhabiting substances, including hydrothermal treatment (Asasutjarit *et al.,* 2007; Sutigno 2000; Ferraz *et al.,* 2011), immersion of lignosellulosic-based materials in different solutions (Ferraz *et al.,* 2012), addition of accelerating agent (Wei *et al.,* 2000; Latorraca *et al.,* 2000; Olorunnisola, 2008; Ferraz *et al.,* 2012), and substitution of part of the cement by silica particles (Del Menezzi *et al.,* 2007).

Giant reed is a perennial herbaceous species that grows in different environments with different ranges of pH, salinity, and drought and trace element bioaccumulator, due to its capacity of absorbing contaminants such as metals without any symptom of stress, especially with phytoremediation processes. The growth of this plant is not inhibited by increasing bauxite (red mud) doses because of alkalinity, salt and metal toxicity, so that it is tolerant of the abiotic stresses and can decontaminate the polluted soil (Alshaal *et al.,* 2013). The presence of bauxite can improve the strength properties and workability of cementituos system due to its Pozzolanic reactivity, reacting with calcium hydroxide and producing additional gel (Soroshian and Won, 1995). According to Ribeiro *et al.* (2013), addition of bauxite changed the hydration process, setting time, and workability, and significantly altered important properties of Portland cement.

It is well known that the hydration temperature and the time duration until this temperature is reached give information on the suitability of a specific species to be bonded with cement (Frybort *et al.,* 2008). In this way, various additives at Nano scale can affect these parameters, such as mineral SiO_2 nanoparticle. As known, calcium silicate hydrate (C-S-H) is the main compound that increases the strength of the concrete paste (Qing *et al.,* 2007). According to Birgisson *et al.* (2012), a small amount of SiO_2 nanoparticles dispersed uniformly in a cement paste makes hydrated products of cement deposit on the nanoparticles due to their higher surface energy, i.e., they act as nucleation sites. Nucleation of hydration products on nanoparticles further promotes and accelerates cement hydration (Lin *et al.,* 2008). As colloidal silica is added, it reacts with the released calcium hydroxide, and tricalcium silicate (C3S) dissolution is accelerated, so that the C-S-H gel in cement+water mixture is formed rapidly (Bjornstrom *et al.,* 2004).

Wood- or lignocellulosic-cement complex is prepared easily and is fabricated from available resources and, therefore, it is widely used in many various types of panel systems. Permanent reduction of wood resources and environmental hazards posed by formaldehyde emission from wood panel products have reached the alarming limit, so that using waste materials is necessary in cement complex manufacturing. In this regard, extensive research is conducted using several types of waste materials. However, none of the researches dealt with the application of abundant giant reeds in cement mixes and improvement of this complex by adding mineral additive at Nano scale.

The aim of the present research was to study the basic strength properties of the giant reed-cement mix. Hence, the effect of using giant reed, as the replacement of the bagasse in cement system, and also of adding SiO_2, as additive at Nano scale to the complex of giant reed particle-cement, on the hydration behavior of cement and properties of the cement-bonded particleboard was examined. Analysis was made of the modulus of rupture (MOR), modulus of elasticity (MOE) and internal bonding (IB) of CBPB by using mathematical model equations (second-order response functions).

2 MATERIAL AND METHODS
2. MATERIJAL I METODE

2.1 Hydration test
2.1. Test hidratacije

Commercial grade Portland cement (ASTM C150, 2009) and hammer-milled bagasse and reed (*Arundo donax* L.) were used to prepare cement pastes. Fifteen cement paste mixtures were designed, batched, and tested to establish the quantitative and qualitative evidence. One control mixture with pure cement was used to have a basis of comparison with other mixtures. Fourteen specimens were batched, cast, and tested with different amounts of NS particles in each mix combined with milled bagasse and reed particles sieved through a 42 wire mesh or without them. For this purpose, different weight ratios were used including 200 g binder with or without NS, 90.50 ml water and 15 g powder of lignocellulosic material for the treatments G, H, I, J, K, L, M, N and O, respectively, and 200 g cement and 90.50 ml distilled water for the treatments A, B, C, D, E and F, respectively (Table 1).

Nanosilica particles were used with specific surface area of 200.1 m^2/g and average particle size of 5-20 nm. Type II commercial grade Portland cement was used in batches and was mixed with Nanosilica at different levels (0, 1.5, 3, 4.5 and 6 %). Distilled water (90.5 ml) was added to the mixture of cement+Nanosilica (200 g) and reed/bagasse (15 g oven dry basis) in a blender and stirred for 3 min. The cement–Nanosilica-reed/bagasse–water mixture was placed in a wide-mouth insulated flask with a thermocouple wire and then it was covered with styrofoam. The flask was sealed with a wrapping tape. The temperature of the mixture was measured and plotted against time. Preliminary work indicated that the hydration temperature started to change after 30 min of testing. The time to attain the maximum temperature was the required setting time of the cement paste mixture. Also, Vicat apparatus was used to obtain the same workability of the mentioned cement pastes by determining initial and final seting times. To determine the effect of adding bagasse and reed particles on the compressive strength and find the correlation between compressive strength and hydration behavior, the mixtures were moulded into one inch cubic stainless steel moulds, and were vibrated on a mechanical vibrator for 4 minutes. The moulds were stored inside a humidity cabinet at 21±3 °C and 100 % RH. Under these conditions, the moulds were preserved for 1, 3, 7 and 28 days. After these periods, they were demoulded and compressive strength test was carried out.

Table 1 Treatments for determination of hydration behavior of cement pastes
Tablica 1. Opis tretmana cementnih pasta za koje je istraženo hidratacijsko ponašanje

Treatment code *Kod tretmana*	**Treatment** *Oznaka*	**Treatment type** *Opis tretmana*
CW	A	cement paste
C1.5	B	cement p.+1.5% NS
C3.0	C	cement p.+3.0% NS
C4.5	D	cement p.+ 4.5% NS
C6.0	E	cement p.+ 6.0% NS
CB	F	cement p.+ bagasse
C1.5B	G	cement p.+1.5% NS+ bagasse
C3.0B	H	cement p.+3.0% NS+ bagasse
C4.5B	I	cement p.+4.5% NS+ bagasse
C6.0B	J	cement p.+6.0% NS+ bagasse
CR	K	cement p.+ reed
C1.5R	L	cement p.+ 1.5% NS+ reed
G3.0R	M	cement p.+ 3.0 NS+ reed
C4.5R	N	cement p.+ 4.5% NS+ reed
C6.0R	O	cement p.+ 6% NS+ reed

2.2 Board preparation and strength measurement
2.2. Priprema ploče i mjerenje čvrstoće

Giant Reed stalks, 3m high, were cut above the water line (collected from a suburb near Zabol City in Sistan-Baloochestan Province of Iran) and split along the grain by a local harvester with the dimensions of 100-200mm (length) × 1-5mm (width) × 0.1-1 mm (thickness). Bagasse particles were purchased from a local market. Crashed reed stalk and bagasse were milled into particles using a hammer mill, and then they were sieved using the sieves with the mesh size >8mm, 6-8, 4-6, 2-4 and <2 mm, separately. The particles were further oven dried to 5 % moisture content (MC) at 90 °C. Commercial Portland cement (type II) was purchased from Sistan Cement Industry Co., Ltd., Iran, to be used as a binder for making panels. CBPBs were produced at nominal board density of 1150 kg/m^3 from the following weight ratios of bagasse/reed particle: 2.55:97.45, 6.94:93.06, 13.38:86.62, 19.81:80.19 and 24.20:75.80. In order to determine the effect of the mineral Nano-particles on the properties of panels, cement was blended with SiO_2 Nano-particles at five levels (0.48, 1.5, 3, 4.5 and 5.52 wt% based on composition by cement weight) in a laboratory mixer with a high rotation speed (2000rpm).The lignocellolosic particle/cement+ Nanosilica/ water weight ratio was set at 1:4:1. Calcium chloride (5 % of cement weight) was dissolved in water and added to the mixture to accelerate cement curing. Particles-cement-water slurry was blended for 10 min in a rotary blender and manually formed into a mat with an approximate moisture content of 20 %. Mat was cold pressed at 4.0 MPa for 48 h. After that, the boards were put into polyethylene bags for 20 days to complete the hydration process as soon possible. Then, they were kept for 6 days in the laboratory to ensure full curing and uniform drying. After 28 days of curing, the panels were trimmed and subjected to the following tests: internal bonding strength and flexural tests.

2.3 Statistical analysis
2.3. Statistička analiza

In this study, response surface methodology (RSM) is used to evaluate the effect of some main process variables and their levels on MOR, MOE and IB values of CBPB. This method finds an appropriate model for predicting the dependent variables as responses. A standard RSM analysis, known as central composite rotatable design (CCRD), is used to create runs according to a logical experimental design and also describe the interaction between the independent variables. The quadratic equation model is used to develop regression equations related to response variable of CBPB production process, as shown by equation (1) below:

$$y = \beta_0 + \sum_{i=1}^{k}\beta_i x_i + \sum_{i<}^{k}\sum_{j}^{k}\beta_{ij} x_i x_j + \sum_{i=1}^{k}\beta_{ii} x_i^2 + \varepsilon.$$

Where x_i and x_j are inputs or independent factors, β_0 is the free term of the equation, coefficients β_1, β_2, β_i

Table 2 Range of process parameters
Tablica 2. Raspon procesnih parametara

Parameters / *Parametri*	**Coded factor** *Kodirani faktor*	**Symbol** *Oznaka*	**Units** *Jedinica*	**Lower limit** *Donja granica*	**Upper limit** *Gornja granica*
Nano-Silica content in cement / *sadržaj nanočestica silicijeva dioksida*	(X_1)	NS	%	1.5	4.5
Particle size of reed and bagasse / *veličina čestica trske i čestica otpada u preradi šećerne trske*	(X_2)	PS	mm	4	8
Weight ratio of bagasse to reed particles / *težinski omjer čestica otpada u preradi šećerne trske i čestica trske*	(X_3)	WR	%	6.94	19.81

Table 3 Experiment design and results
Tablica 3. Dizajn eksperimenta i rezultati

Run	**Coded values** / *Kodirane vrijednosti*			**Actual values** / *Stvarne vrijednosti*			MOR	MOE	IB
	X_1	X_2	X_3	NS	PS	WR	MPa	MPa	MPa
1	1	1	1	4.50	8.00	19.81	12.45	2350	0.43
2	0	0	1.68	3.00	6.00	24.20	16.34	2780	0.65
3	-1.68	0	0	0.48	6.00	13.38	7.9	1367	0.21
4	-1	1	-1	1.50	8.00	6.94	4.9	789	0.18
5	1.68	0	0	5.52	6.00	13.38	11.56	2246	0.33
6	1	-1	-1	4.50	4.00	6.94	9.6	1678	0.25
7	-1	-1	-1	1.50	4.00	6.94	6.6	845	0.2
8	0	0	0	3.00	6.00	13.38	11.2	2050	0.47
9	0	-1.68	0	3.00	2.64	13.38	8.8	1456	0.26
10	1	-1	1	4.50	4.00	19.81	12	2456	0.52
11	0	1.68	0	3.00	9.36	13.38	3.75	456	0.19
12	0	0	0	3.00	6.00	13.38	10.87	1998	0.48
13	0	0	0	3.00	6.00	13.38	11	2089	0.49
14	0	0	-1.68	3.00	6.00	2.55	6.65	768	0.32
15	0	0	0	3.00	6.00	13.38	12	2134	0.495
16	-1	-1	1	1.50	4.00	19.81	8.56	1546	0.42
17	0	0	0	3.00	6.00	13.38	9.98	1867	0.493
18	-1	1	1	1.50	8.00	19.81	9.56	1784	0.32
19	1	1	-1	4.50	8.00	6.94	6	589	0.22
20	0	0	0	3.00	6.00	13.38	11.88	1879	0.5

are linear terms; β_{11}, β_{22}, β_{ii} are quadratic terms; β_{12}, β_{13}, $\beta_{i-1,j}$ are the interaction terms and ϵ denotes random error.

The CCRD offers n^2 factorial runs, $2n$ axial runs and n center runs (six replicates), with n as number of variables. The axial points were added to estimate the quadratic terms of the model and collected at (±α, 0, 0), (0, ±α, 0), and (0, 0, ±α). α is defined depending on the region of operability and region of interest. In this research, α value was selected as 1.68, and 20 experimental design points were considered including 6 center points. It was assumed that the design is rotatable when the value of α is determined. Table 2 shows three production parameters, i.e. Nano-Silica content in cement (X_1), size of reed and bagasse particles (X_2) and weight ratio of bagasse to reed particles (X_3) and their five levels.

A total of 20 experiments were required according to the CCRD design. The sequence of the experiment was randomized to minimize the effect of the uncontrolled factor (Table 3). For evaluating the statistical significance of the generated regression model, the analysis of variance (ANOVA) for the model was also performed at 5 % significance level incorporated in Expert Design software.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

3.1 Hydration temperature
3.1. Temperatura hidratacije

In order to study the effect of the NS content as additive on hydration of cement based composites, the hydration temperature of the pure cement paste, cement-bagasse-based- and cement-reed-based-composites containing different levels of NS particles was monitored by isothermal calorimetric analysis. As shown in Figs. 1, 2 and 3, there is a significant difference between the heat of the hydration of one gram cement evolved during its hydration, cement-bagasse and cement-reed particles during the first 12 hours at a constant water to cement ratio.

While 6 wt.% additives decreased the hydration temperature, hydration temperature curves indicated that the addition of 1.5-4.5 wt.% NS particles to pure cement resulted in an increase of T_{max} in all samples (Figure 1). Moreover, the addition of NS shortened the initial and final setting times of the paste compared to the initial and final setting times of pure cement paste. When the hydration process begins, hydrate products diffuse and coat nanoparticles so that the cement hy-

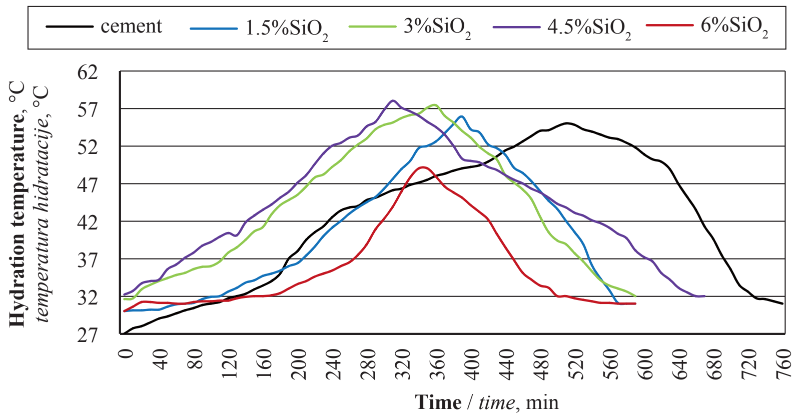

Figure 1 Exothermic curves of NS–cement mixtures as compared to neat cement
Slika 1. Egzotermne krivulje smjesa cementa s česticama NS-a u usporedbi s čistim cementom

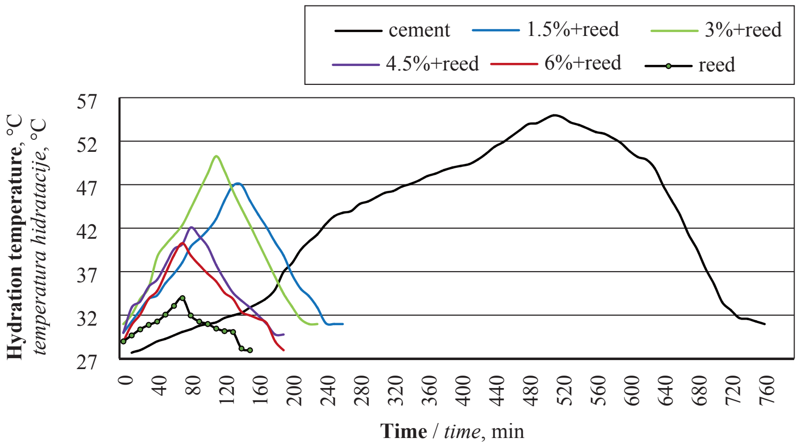

Figure 2 Exothermic curves of reed particles+NS+cement mixtures as compared to neat cement
Slika 2. Egzotermne krivulje smjesa cementa s česticama NS-a i trske u usporedbi s čistim cementom

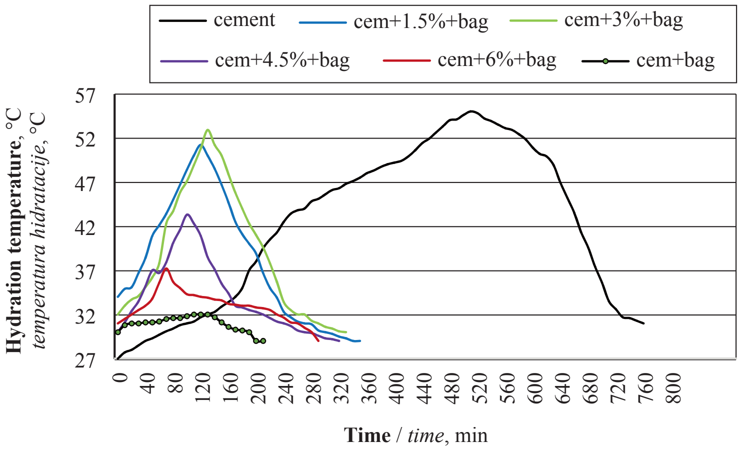

Figure 3 Exothermic curves of bagasse particles+NS+cement mixtures as compared to neat cement
Slika 3. Egzotermne krivulje smjesa cementa s česticama NS-a i česticama otpada u preradi šećerne trske u usporedbi s čistim cementom

dration speed rises, and the cement paste becomes more homogeneous and compact (Jalal *et al.,* 2012). Besides, the time of reaching the main rate peak (t_{max}) changes significantly due to more pozzolanic reaction creating an earlier peak. Therefore, with an increase of the Nanosilica amount from 3 to 4.5%, more reactive nuclei are created during the hydration, and t_{max} decreases correspondingly. However, the values of water absorption and apparent porosity of Nanosilica particles are high (Senff *et al.,* 2010) so that the water to binder ratio becomes low due to the absorption process. As a result, a large amount of cement particles are still dehydrated at the end of cement hydration process in the sample containing 6% Nano-silica. Hence, although Nano-particles can accelerate cement hydration to a great extent in the early ages, the later hydration of cement is hindered.

In cement+Nano-Silica + reed/bagasse mixture, the addition of 3 % Nanosilica increased maximum heat of hydration (Figures 2 and 3). Compared to the analysis results of pure cement samples (510 min), the induction period reduced to 110 min for reed component (Figure 2) and 130 min for bagasse component, respectively (Figure 3), when 3 wt.% cement was replaced by Nanosilica. Besides, during the hydration, all of the cement samples that contained reed or bagasse showed lower maximum hydration temperature in comparison with pure sample or samples containing only Nanosilica as an additive. This is consistent with the results obtained by (Bilba *et al.,* 2003; Xie *et al.,* 2016), which showed that hemicellulose and lignin in plant fiber component have a negative effect on cement hydration process. Moreover, this phenomenon is related to the partial substitution of cement with lignocellulosic particles, causing excessive use of water and absorption of a part of the water for hydration. Sudin and Swamy (2006) and Alpar et al. (2011) stipulated that the delayed setting time of Portland cement matrix was caused by high content of carbohydrates, such as sugars in the fiber. The dissolution of these soluble sugar compounds forms calcium mixtures in the cement paste. These mixtures decrease hydration temperature of cement matrix and delay the formation of hydration products. It was also observed that using bagasse prolonged the initial and final setting times and raised the T_{max} of the paste, compared to the initial and final setting times and T_{max} of cement paste containing reed.

3.2 Vicat test and compressive strength
3.2. *Vicat* test i tlačna čvrstoća

The influence of reed and bagasse on the initial and final setting times of the pure cement paste, 1.5 %, 3 %, 4.5 % and 6 % SiO_2 Nano-particle systems are shown in Figure 4.

The results indicate that an increased level of SiO_2 replacement results in shortened setting time. Shortening effect is probably due to higher volume fraction of Nano-particles, higher specific surface area in comparison with cement, and hence more absorption of water by these particles. Moreover, adding NS particles to hydrating cement increases formation of calcium silicate hydrate (C–S–H) gel due to reaction of Nano-SiO_2 with $Ca(OH)_2$ (calcium hydroxide, CH), accelerates the hydration of tricalcium silicate (C_3S) and dicalcium silicate (C_2S) and fills pores in the C–S–H crystal net (Biricik and Sarier, 2014; Senff *et al.,* 2009). Then, SiO_2 decreases the setting time of the cement paste and reduces water leakage, while improving the cohesiveness of fresh cement mixtures (Senff *et al.,* 2009).

Results also showed that using reed particles increases initial and final setting times of cement with or without NS, while bagasse particles reduce initial and final setting times of the cement mixture. This is likely because the addition of bagasse particles in cement increases the water demand to obtain a plastic mix of cement due to its spongy structure; however, the addition of reed particles minimizes water demand to obtain a plastic mix of the cement due to the existence of smooth outer surface and also hydrophobic waxy layer coating the outer surface of reed stalks. The presumable reason behind this phenomenon is the decrease in fluidity and increase in stiffness of cement that in-

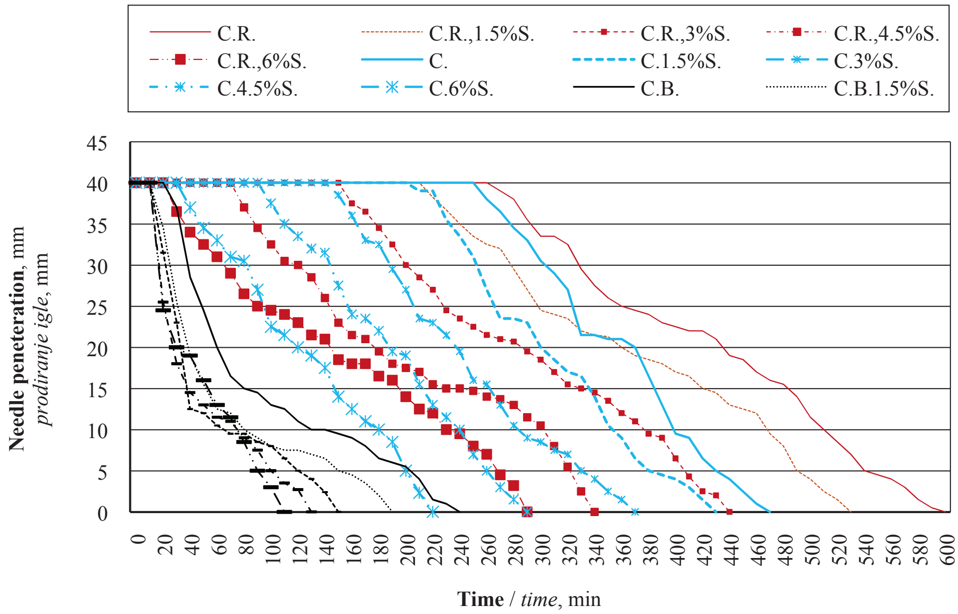

Figure 4 Initial and final hydration curves of lignocellulosic particles+ NS+ cement mixtures as compared to neat cement
Slika 4. Krivulje početne i završne hidratacije različitih smjesa cementa s česticama lignoceluloznog materijala i nanočesticama silicijeva dioksida u usporedbi s čistim cementom

crease the water demand due to higher absorption of water by hygroscopic particles (Byung-Wan *et al.,* 2014). However, the reed exerts a smoothing effect on the cement particles, thus decreasing the interior attrition coefficient, which in turn promotes the fluidity of cement paste.

According to Figure 5, not only additives (NS) but also lignocellulosic content differently influenced the compressive strength of cementitious samples during different periods. The addition of NS from 0 to 6 % significantly increased the compressive strength of the pure cement paste in the hardening stages (after 1, 3 and 7 days), while this variable decreased after 28 days for samples containing more than 4.5 % of NS. This is related to hydrophilic characteristics of silicates and silica gel formation, which strongly bound to portlandite compound formed in the early ages of cement paste hydration. Due to the excess NS content and the subsequent high water absorption of the NS and formation of silica gel, hardening process of the paste is accelerated at early ages (as shown in Figure 5), while a loose coagulation of cementitious structure may form at later ages, because of the lack of enough water for completion of cement particles hydration; so the strength of samples decreases (Kotsay, 2013).

Besides, due to the addition of a high amount of Nano-silica to cement complex and the resulting increase in its viscosity, a large amount of air can be trapped into the system increasing the porosity of hardened concrete (Yu *et al.,* 2014). In the presence of the optimal amount of Nanosilica, the resulting positive effect of the nucleation and the negative influence of the entrapped air can be equal. Therefore, using a specified content of Nano silica, the porosity of the hardened cement can be decreased.

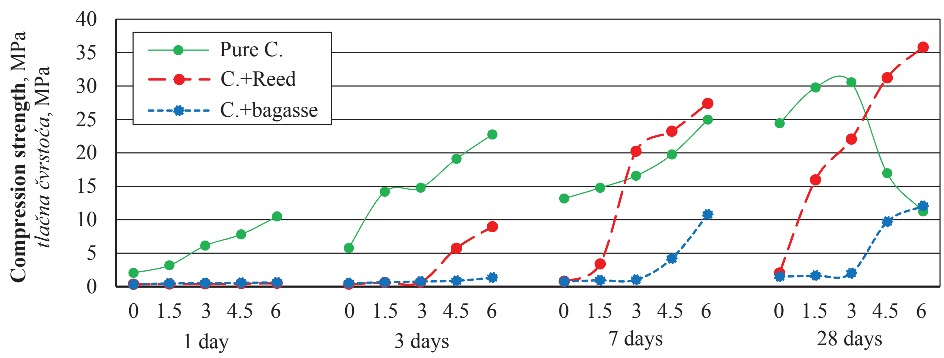

Figure 5 Compression strength development in pure cement pastes, reed+cement paste and bagasse+cement paste during 1 day, 3 days, 7 days and 28 days of hydration
Slika 5. Promjena tlačne čvrstoće čiste cementne paste, cementne paste s česticama trske i cementne paste s česticama otpada u preradi šećerne trske tijekom jednog dana, 3 dana, 7 dana i 28 dana hidratacije

3.3 Mechanical properties of CBPB
3.3. Mehanička svojstva cementnih iverica

Mechanical properties were optimized by the response surface methodology (RSM). The CCRD was used to develop the correlation between the process variables, including Nanosilica content (NS), reed and bagasse particle size (PS) and weight ratio of bagasse/reed particles (WR) (coded as X_1, X_2, and X_3, respectively) and responses, MOR, MOE and IB. The quadratic models of the responses are presented in Equations (2, 3 and 4) in terms of the coded factors according to their significance:

The results of the analysis of variance (ANOVA) for quadratic models are shown in Table 4 for MOR, MOE and IB. According to Table (4), the weight ratio of R/B particles (WR) is the most important factor affecting not only MOR but also MOE and IB, followed by Nano silica content (NS) and particle size (PS) (because F values of WR are higher than NS and PS for all responses). Besides, interaction effect of PS and WR (X_2X_3) on MOR, MOE and IB, NS and PS (X_1X_2) on MOE, and NS and WR (X_1X_3) on IB are significant; however, the effect of the quadratic value of WR (X_3^2) on MOR and IB and NS (X_1^2) and WR (X_3^2) are not significant. Coefficients of determination (R^2) for MOR, MOE and IB showed that 93.4 %, 94.6 % and 99.7 of all variations are explained by the model, respectively.

R^2 values obtained after adjusting the terms of the model for MOR, MOE and IB are 90.4 %, 92.1 % and 99.5 %, respectively. The comparison of R^2_{Adj} = 0.9040, 0.9214 and 0.9952 with R^2_{Pred}=0.8053, 0.8190 and 0.9916 shows that both terms are in good agreement with each other and the models can explain 80.53 %, 81.90 % and 99.16 % variance of the new data.

The "Lack of Fit F-value" of 0.237, 0.07 and 0.74 for MOR, MOE and IB, respectively, imply that the Lack of Fit is not significant relative to the pure error. Hence, the models should fit the data. Improved precision and reliability of test results are shown below the values of coefficient of variation (C.V.) for MOR, MOE and IB; they are 9.73 %, 11.28 % and 2.6 %, respectively.

The influence of three factors including NS, PS and WR are shown in three-dimensional response of contour (Figures 6, 7 and 8). Figure 6, 7 (left) and 8 (left) illustrate the effect of two variables including PS

Table 4 Analysis of variance of MOR, MOE and IB
Tablica 4. Analiza varijance MOR-a, MOE-a i IB-a

Source / *Izvor varijacije*		**Sum of squares** *Zbroj kvadrata*	***df***	**Mean Square** *Srednja vrijednost kvadrata*	***F* Value** *F-vrijednost*	***p*-Value** *p-vrijednost* prob > F	**Sig.**
Model:		160.67 7.991E+006	6 6	26.78 1.332E+006	30.81 38.14	< 0.0001 < 0.0001	** **
MOR		160.67	6	26.78	30.81	< 0.0001	**
MOE		7991	6	61332	38.14	<0.0001	**
IB		0.37	7	0.018	197.24	<0.0001	**
X_1	MOR	20.14	1	20.14	23.17	0.0003	**
	MOE	9.423E+005	1	9.423E+005	26.99	0.0002	**
	IB	0.018	1	0.018	197.24	< 0.0001	**
X_2	MOR	11.16	1	11.16	12.83	0.0033	**
	MOE	5.317E+005	1	5.317E+005	15.23	0.0018	**
	IB	9.370E-003	1	9.370E-003	100.23	< 0.0001	**
X_3	MOR	73.89	1	73.89	85.01	< 0.0001	**
	MOE	4.250E+006	1	4.250E+006	121.73	< 0.0001	**
	IB	0.14	1	0.14	1524.21	< 0.0001	**
X_1^2	MOR	4.89	1	4.89	5.63	0.0338	*
	IB	0.086	1	0.086	919.53	< 0.0001	**
X_2^2	MOR	47.23	1	47.23	54.33	< 0.0001	**
	MOE	1.825E+006	1	1.825E+006	52.28	< 0.0001	**
	IB	0.13	1	0.13	1339.66	< 0.0001	**
AB	MOE	2.370E+005	1	2.370E+005	6.79	0.0218	*
BC	MOR	5.70	1	5.70	6.55	0.0238	*
	MOE	2.038E+005	1	2.038E+005	5.84	0.0311	*
	IB	2.450E-003	1	2.450E-003	26.21	0.0003	**
AC	IB	1.800E-003	1	1.800E-003	19.25	0.0009	**
MOR	Lack of Fit	8.57	8	1.07	1.96	0.2370	ns
MOE	Lack of Fit	3.932E+005	8	49155.15	4.05	0.0700	ns
IB	Lack of Fit	5.118E-004	7	7.312E-005	0.60	0.7405	ns
	MOR			MOE		IB	
Std. Dev.=0.93, R^2=0.9343, Adj R^2=0.9040, C.V.= 9.73 Pred R^2=0.8053,			Std. Dev.=186.86, R^2=0.9462, Adj R^2=0.9214,C.V.=11.28, Pred R^2=0.8190			Std. Dev.=9.669E-003, R^2=0.9970, Adj R^2=0.9952, C.V.=2.6, Pred R^2=0.9916	

(X_2) and WR (X_3) on MOR, MOE and IB when NS (X_1) is held at center level. MOR, MOE and IB increase as bagasse content increases at both (i.e. lower and higher) values of PS. Maximum MOR, MOE and IB are achieved at maximum level of WR (>19.81 %) and 6 mm PS.

The boards with the highest content of reed particles had the lowest MOR and MOE values. The outer surface of reed is believed to be richly covered by silica and wax (Perdue *et al.,* 1958). Smooth, hard and waxy surface of these types of lignocellulosic material may be one of the likely reasons of difficulty and failure of adhesion between the cement and reed particles.

With hydration of cement, the metal-hydroxyl groups, such as -Ca-OH, -Si- OH, -Al-OH and Fe-OH (due to hydration and hydrolysis of silicates, aluminates and to a lesser extent ferrites of calcium in the cement paste) are present at the surface of reed particles to form chemical bonding; however, according to Wei and Tomita (2001), the bonding strength does not benefit from the presence of silica at the surface of lignocellulosic particles. According to observations during the IB test, the adhesive disconnection mainly took place between hardened cement and reed particles rather than on bagasse particles. In fact, the presence of wax and surface properties of reed particles may affect adversely the bonding of CBPB.

It was determined that the content of silica and lignin in the reed (1.18-1.97 % and 25 %, respectively) (Wang *et al.,* 2013) is higher than that of bagasse particles 0.98 % and 21 %, respectively (Agnihotri *et al.,* 2010). Increasing lignin content might contribute to a higher compressive strength and hardness values (as shown in Figure 5) and consequently brittleness. Higher silica content in reed stalks results in higher stiffness and lower flexibility (Wu *et al.,* 2010), simultaneously. This means that the compaction ratio of panels and

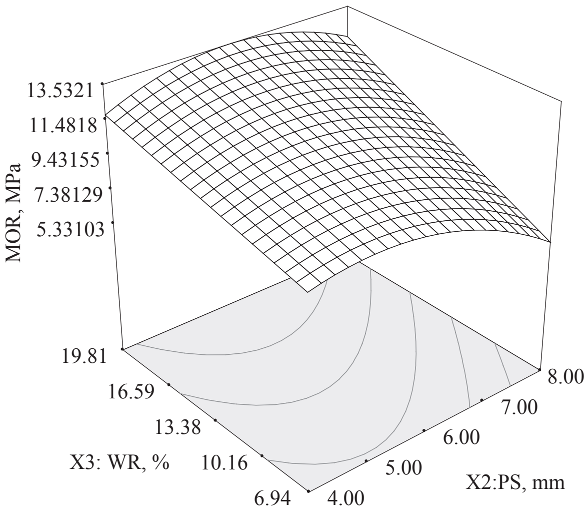

Figure 6 Three dimensional surface plots predicting MOR from the equation model: effect of particle size and weight ratio of bagasse to reed particles at center level of Nano SiO_2 content
Slika 6. Prikaz trodimenzionalnih površina koje predviđaju MOR iz jednadžbe modela: učinak veličine čestica i težinskog omjera čestica otpada u preradi šećerne trske i čestica trske na središnjoj razini sadržaja nanočestica silicijeva dioksida

consequently the contact between particles during pressing decrease, so the bending strength decreases.

Figure 7 (right) shows 3D surface graphs for the interaction effects of NS content and particle size (X_1X_2) on MOE. It can be seen that maximum value of MOE is achieved with the combination of highest NS and almost center level of PS (at 5.52 % NS and 6-5.25 mm particle size). Increasing NS at both (i.e. lower and higher) values of PS, MOE values increased, but as it can be seen in Figure 7 (right) and as noted in the ANOVA table, increase in NS has more influence on the increase in MOE.

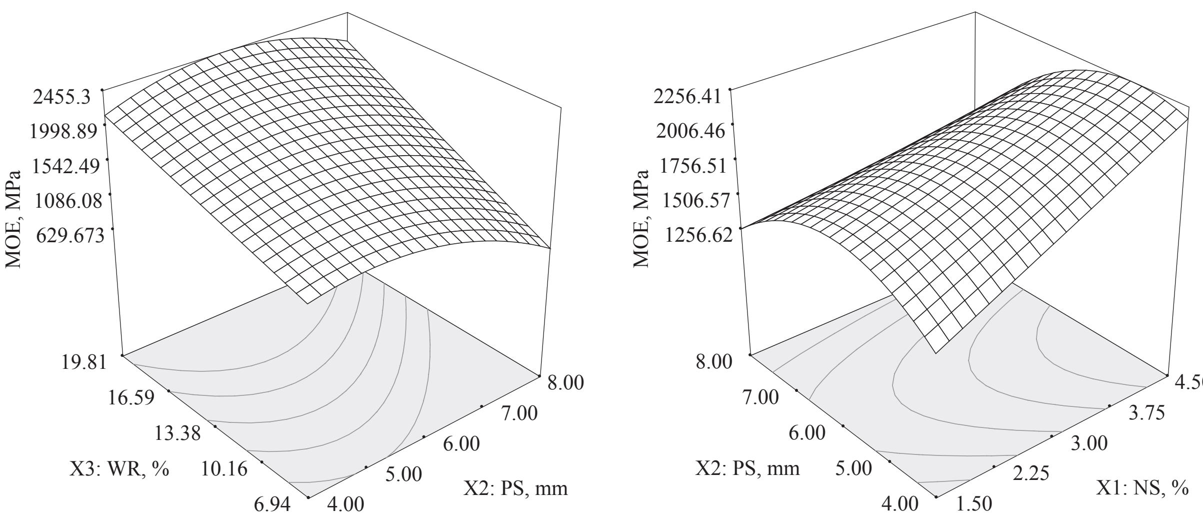

Figure 7 Three dimensional surface plots predicting MOE from the equation model: effect of particle size and weight ratio of bagasse to reed particles at center level of NS content (left); effect of NS content and particle size at center level of weight ratio of bagasse to reed particles (right)
Slika 7. Prikaz trodimenzionalnih površina koje predviđaju MOE iz jednadžbe modela: učinak veličine čestica i težinskog omjera čestica otpada u preradi šećerne trske i čestica trske na središnjoj razini sadržaja nanočestica silicijeva dioksida (lijevo); učinak sadržaja nanočestica silicijeva dioksida i veličine čestica na središnjoj razini omjera čestica otpada u preradi šećerne trske i čestica trske (desno)

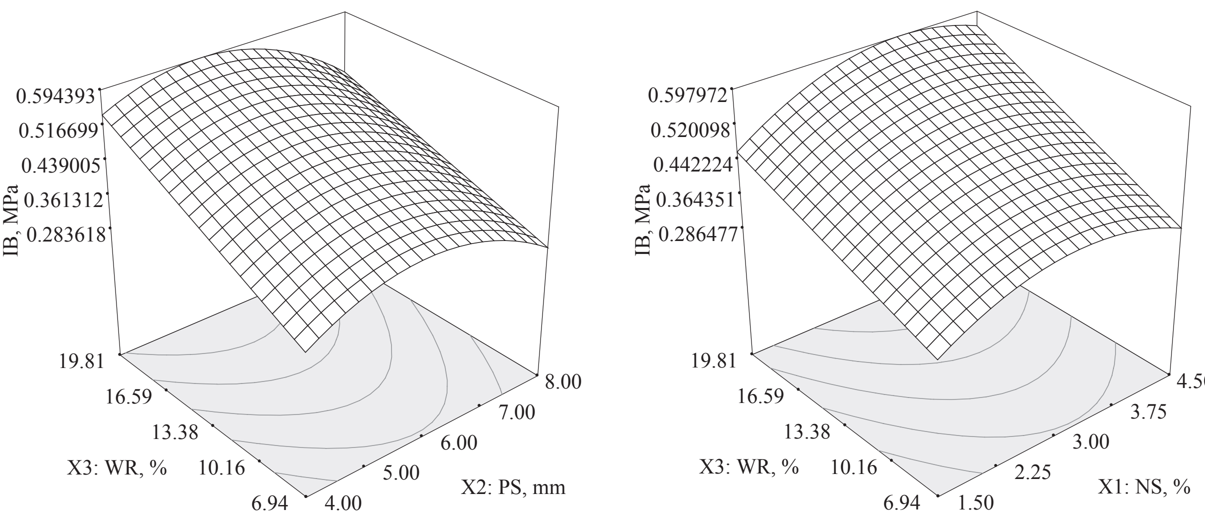

Figure 8 Three dimensional surface plots predicting IB from the equation model: effect of particle size and weight ratio of bagasse to reed particles at center level of NS content (left); effect of NS content and weight ratio of bagasse to reed particles at center level of particle size (right)

Slika 8. Prikaz trodimenzionalnih površina koje predviđaju IB iz jednadžbe modela: učinak veličine čestica i težinskog omjera čestica otpada u preradi šećerne trske i čestica trske na središnjoj razini sadržaja nanočestica silicijeva dioksida (lijevo); učinak sadržaja nanočestica silicijeva dioksida i omjera čestica otpada u preradi šećerne trske i čestica trske na središnjoj razini veličine čestica (desno)

Due to using NS, important effects for the hydration kinetics and the microstructure of the cement paste are revealed, such as (a) an increase in the initial hydration rate (as shown in Figures 1 and 2), (b) an increase of the amount of C-S-H gel in the paste through pozzolanic reaction due to the reaction of NS with CH during the hydration process, (c) reduction of porosity through the pore size refinement in the early ages, so NS acted as a core that strongly sticks to the hydrated cement, and finally, (d) improvement in the mechanical properties of the C-S-H gel itself by increasing the average chain length of C-S-H gel (Gaitero *et al.,* 2010). Besides, addition of additives as a pozzolanic mineral to cement mixtures decreases the inhibitory influence of extractives. Due to higher specific surface area of these additives than cement and higher sorption of these materials, the adsorption of water-soluble extractives occurs on the surface of NS first, while the concentration of extractives and their negative effects on hydration process decreases.

The influence of varying two factors of NS and WR (X_1X_3) on IB at a constant particle size, i.e. 6 mm, is depicted in Figure 8 (right). It can be observed from the figure that higher WR at both (i.e. lower and higher) values of NS results in lower IB. Since a large amount of cement has already been replaced by NS powder, the water to cement ratio is relatively stable. However, the water amount of samples containing high level of NS is still relatively low. Since the water used can be significantly absorbed by the hydrophilic materials, the amount of hydrated cement particles is fixed at lower limits. Finally, to further decrease the IB of the CBPB around 4.5 % and higher, NS is added into the lignocellulosic particles-cement matrix. On the other hand, 0.48 % to 3 % NS increased IB despite the increased demand for water in the matrix. In fact, Nano-scale SiO_2 plays a role not only as a filler to improve microstructure (which is a factor affecting the increase in cohesiveness of the paste and IB), but also as an accelerator of pozzolanic reaction in cement matrix (Qing *et al.,* 2007; Jo *et al.,* 2007).

4 CONCLUSION
4. ZAKLJUČAK

More effective utilization of wood and forest resources and uses of agricultural products in many valuable fields can be achieved by using reed and bagasse as lignocellulosic sources and alternative raw materials in cement-bonded particleboard industry. Thus, the effect of Nanosilica content, particle size of bagasse and reed and weight ratio of bagasse to reed particle were evaluated using RSM model. The major conclusions based on the data obtained in this paper can be summarized as follows:

1. NS makes cement paste thicker and accelerates the cement hydration process, while addition of lignosellulosic particles remarkably delayed the hydration process of the cement paste with or without NS.
2. Addition of reed and bagasse into the cement past had positive effect on the compressive strengths of hardened cement, especially at the end of the hydration ages.
3. While MOR, MOE and IB increased with increasing the particle size of bagasse and reed to a certain value and then decreased as the particle size of bagasse and reed increased more than a certain value, as the weight ratio of bagasse to reed increased, MOR, MOE and IB increased directly. Moreover, as NS content increased, MOE increased directly; however, IB enhanced as NS content increased to a certain value and then it decreased as NS content increased more than the certain value.
4. The mathematical model of MOR, MOE and IB developed by RSM presents desirable information with a small number of experimentations. The mod-

el is rationally appropriate and can predict the values of responses within the studied limit of parameters. It is determined from ANOVA that WR has a maximum effect on MOR, MOE and IB compared to other selected variables.

5. It is also concluded from the ANOVA that the developed model can be effectively used to predict the MOR, MOE and IB of the CBPB at 95 % confidence level. The values of R^2 and adjusted R^2 are 93.43 % and 90.40 % for MOR, 94.62 % and 92.14 % for MOE, and 99.70 % and 99.52 % for IB, respectively, and hence, the repeatability of the results is reasonable.

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