Effect of Dry Heat and Polishing Treatment on the Germination Rate and Performance of Seeds from *Cassia obtusifolia* L. as a Turf Filling Material

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Seeds from *Cassia obtusifolia* L., *i.e. Semen cassiae* (SC) were evaluated as environmentally friendly filler particles for artificial turf. The goal was to avoid unwanted germination problems of SC under wet conditions. This work evaluated the influence of different pretreatments on the germination rate and performance of SC. After the combination of polishing and dry heat (90 °C, 72 h) treatment, the germination rate of SC decreased to 0% and the activation energy increased to 216 kJ/mol. Compared with the untreated SC, the thermal stability of SC improved, with an initial degradation temperature of 214 °C and a pyrolysis residue of 31.9%. Additionally, the resilience and the water absorption of SC as a filler material increased to 4.13% and 169%, respectively. This study provides an effective pretreatment method for the solution of germination problem and the performance improvement of SC. This makes the pretreated SC a prospective candidate as an environmentally protective granular filling material.

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INTRODUCTION

The artificial turf of a football field needs to be filled with suitable particles to maintain good performance and protect the safety of athletes (Xue 2021). Currently, the commonly used filling particles mainly include rubber particles SBR, EPDM, TPE, *etc*., and the particle size is usually between 0.5 and 2.5 mm. All of these can be categorized as micro-plastic particles, and they will be continuously lost in the process of use, and they will need to be continuously supplemented (Liu *et al.* 2021). The particles that are lost can be carried by drainage systems into oceans, causing micro-plastic pollution (Rezania *et al.* 2018). As the problem of micro-plastic pollution becomes increasingly serious, more measures are being taken to ban plastic and limit plastic around the world (Zhang and Wei 2021). Among them, the European Commission is considering a ban on these kinds of tiny plastic particles from being used in artificial turf. Therefore, it is particularly important and urgent to find green particles to replace micro-plastic particles. Environmentally friendly artificial turf filling materials can be obtained by the processing of waste materials in such a way as to cause no environmental hazards. Thus, the waste materials can be formed into biodegradable products for reuse. The environment-friendly filler particles for artificial turf need to have good performance, such as thermal properties, resilience, and water absorption, to replace the existing rubber filler particles of artificial turf. *Semen cassiae* (SC) is the formal name of dry mature seeds of the plant *Cassia obtusifolia* L. Such seeds have been used in artificial turf filling granules at a production company instead of rubber particles (CCGRASS Co., LTD, Jiangsu, China). The seeds have been employed as a buffer layer to straighten the synthetic lawn thread. However, the SC will germinate under moist conditions, and this affects its durability as filler particles (Xie *et al.* 2009). For example, the outdoor artificial turf is susceptible to rainy weather that requires regular replacement of filling particles, and the performance of SC will be different when it is exposed to a humid environment for a long time. Therefore, the solution of germination problem is a precondition so that SC can be used as particle filling material for long periods.

Moreno *et al.* (2019) demonstrated that an increase in the drying temperature caused a decrease in the consumption of energy required to dry corn seeds. They found that a drying temperature over 75 °C could significantly reduce the germination of the corn seeds. Falconí and Yánez-Mendizábal (2016) reported that the germination rate of seeds decreased with the increase of treatment time after the heat treatment of 12 h. Therefore, dry heat treatment temperature above 70 °C and a time more than 24 h could be an effective treatment to reduce the germination rate of SC (Pereira and Ferreira 2010; Merou *et al.* 2011; Rodrigues-Junior *et al.* 2016; Nie *et al.* 2017; Huang *et al.* 2020). The activation energy is the minimum amount of energy required to initiate a reaction, which was one of the indexes for appraising the difficulty degree of a reaction. The chemical reaction rate of seed germination has been found to be related closely to the value of the activation energy (Jiao *et al.* 2014; Edreis and Yao 2016; Wang *et al.* 2017; Xu *et al.* 2020). Hara *et al.* (2005) showed that calculating the activation energy of temperature-dependent reactions could be used to study germination rates.

The purpose of this research was to reduce the germination rate of SC to meet the requirements of its use as an environmentally friendly artificial turf filling particles. This study compared the effect of dry heat treatment on the germination rate of untreated and polished SC, and the activation energy was calculated to analyze the germination kinetics of SC after different treatments. The best-performing treatments on the thermodynamic performance, water absorption of, and resilience of SC as artificial turf filling material was also evaluated.

EXPERIMENTAL

Materials

The untreated SC was obtained from Nanjing, Jiangsu Province, China. The polished SC was processed from Ningbo, Zhejiang Province, China, were purchased from a local toy material store.

Different Treatments for SC

For the dry heat treatment, the SC was heated in an oven (DHG-9140A; Shanghai Huitai Instrument Manufacturing Co., Shanghai, China) at a constant temperature of 70, 80, or 90 °C for 24, 48, or 72 h.

For the polishing treatment, the surfaces of the SC were polished by a roller type polishing machine (PGJ-1S; Xinxiang SLT Mechanical Equipment Co., Xinxiang, China). The rotating axis in the roller made the seeds perform displacement movement and rub against the cotton cloth fixed on the spiral axis until the surface of the seeds was smooth.

For the combination of polishing and dry heat treatment, the polished SC were dry-heat treated with the same parameters as above.

As shown in Fig. 1, the processed SC were stored in a polyethylene terephthalate (PET) sealed jar for later use.

Fig. 1. Image of the 4 kinds of SC samples

Seed-germination Determination

The germination test was carried out on three replicates of 50 seeds from each treatment placed on moist filter paper in square Petri dishes with a side length of 13 cm. The seeds were cultured in a constant temperature and humidity seed incubator (HZ-2004G; Dongguan Lixian Instrument Technology Co., Dongguan, China). The seeds were cultured according to the best temperature and humidity for seed germination, at 23 °C and 80%, respectively. The seeds were kept moist during the germination test, and the germination rate was recorded every 24 h (8 h in light and 16 h in dark) until it remained unchanged for 72 h (Shen *et al.* 2008; Merou *et al.* 2011). Figure 2 illustrates the germination rate recording process. SPSS (IBM SPSS Statistics 26.lnk, Armonk, NY) was used to analyze the significance of germination rate after different pretreatment.

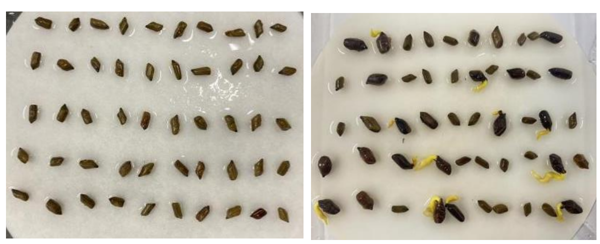

Fig. 2. Images of the germination rate process of SC

Thermal Analysis

The thermogravimetric (TG) curve of the SC was determined by a chip analyzer (STA449 F3; Netzsch, Shanghai, China). The sample was a single seed weighing between 10 and 20 mg, and the heating rate was 20 °C/min. The purge gas and protective gas rate was 20 mL/min, and the temperature range was 30 to 800 °C.

Kinetic Analysis

To explore the thermal decomposition reaction kinetics of thermal decomposition processes of the SC seeds after different treatments, Ozawa's method was mainly used, as seen in Eq. 1,

$$\log\beta = \log\frac{AE}{RF(\alpha)} - 2.315 - 0.4567\frac{E}{RT} \tag{1}$$

where α is the conversion rate, R is the gas constant (J/mol·K), β is the heating rate (K/min), T is the temperature at the highest rate of heating (K), A is the per-exponential constant (s^{-1}), E is the activation energy (kJ/mol), and $F(\alpha)$ is the functional integration of the decomposition reaction mechanism. When the α values were the same, log(β) showed a linear relationship with 1,000/T. The activation energy could be calculated according to the linear slope (Edreis and Yao 2016; Wang *et al.* 2017).

Microscopic Morphology

The surface of the SC was treated by spraying gold with an ion sputtering instrument for 1 min, and the surface microscopic morphology of the SC was observed with a field emission scanning electron microscope (S-4800; Hitachi, Tokyo, Japan).

Resilience

The SC was placed in a cylindrical filling mold to be filled and compacted to simulate the force conditions when used as a granular filling material, with a diameter of 5.5 cm and a filling thickness of 1.5 cm (Fig. 3). And the sample was specially used for the resilience test of the filled materials, which was tested by the KD4088 rubber impact resilience testing machine (Kaide Test Machinery Co., Yangzhou, China), as can be seen in Fig. 3b. The height of the pendulum rebound indicated the size of the resilience rate when the specimen was impacted.

Fig. 3. The SC samples for testing the resilience

Water Absorption

The 24 h water absorption of the SC treated with different treatments was measured according to the GB/T 1462 (2005) standard. The mass before water absorption (m1) and the mass after 24 h of water absorption (m2) were weighed. The calculation formula of the water absorption can be seen in Eq. 2,

$$w = \frac{m2 - m1}{m1} \quad (2)$$

where w is the water absorption (%).

RESULTS AND DISCUSSION

Seed Germination

In most treatments, SC displayed a low germination rate and differed significantly from the control (Merou *et al.* 2011). The germination rate of SC remained unchanged after 7 d of culture, so the final germination rate was recorded for a 7 d cycle (Fig. 4). The SC dry-heating treatment was unsuccessful in breaking dormancy, with the germination percentage ranging between 3.33% and 39.33%, and all the treatments were lower than the control (Table 1). According to the existing research, the SC activity began to decrease at the dry heat treatment temperature of 70 °C (Falconí and Yánez-Mendizábal 2016; Zhang *et al.* 2017). Among them, the germination was lowest when the temperature was 90 °C and the treatment time was 48 h, which was 3.33% (Table 1). In addition, the polishing treatment also resulted in a lower germination percentage of 14% (Table 2) (Pereira and Ferreira 2010). After polishing and dry-heating at 90 °C for 72 h, the SC did not germinate at all (Table 2). At this point, it was likely that the SC activity was reduced or that mortality occurred (Rodrigues-Junior *et al.* 2016; Nie *et al.* 2017; Huang *et al.* 2020). In conclusion, three effective treatments were selected under different treatment temperatures and times (Fig. 4), which will be discussed in the following experiments.

Table 1. The Germination Rate of the Dry Heat-treated SC

Untreated	**52.67% ± 3.06^{a}**		
(Dry-heated) Time / Temperature	24 h	48 h	72 h
70 °C	23.33% ± 5.77^{c}	24.67% ± 3.05^{c}	39.33% ± 3.05^{b}
80 °C	10.00% ± 2.00^{b}	10.00% ± 3.46^{b}	13.33% ± 2.31^{b}
90 °C	4.67% ± 1.15^{bc}	3.33% ± 1.15^{c}	8.00% ± 2.00^{b}
Different letters in the same column represent significant differences (*P*<0.05)			

Table 2. The Germination Rate of the SC Treated by the Combination of Polishing and Dry Heat

Polished	**14.00 ± 5.29^{b}**		
(Combined) Time / Temperature	24 h	48 h	72 h
70 °C	1.33% ± 1.15^{c}	1.33% ± 1.15^{c}	0.67% ± 1.15^{c}
80 °C	1.33% ± 1.15^{c}	1.33% ± 1.15^{c}	0.67% ± 1.15^{c}
90 °C	0	0	0
Different letters in the same column represent significant differences (*P*<0.05)			

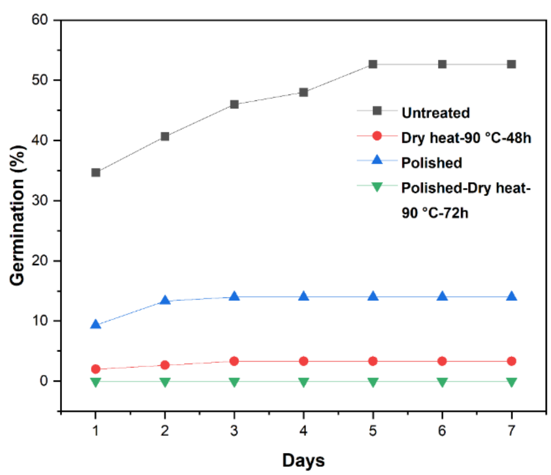

Fig. 4. Growth curve of the germination rate of SC

Thermal Analysis

The TG/derivative thermogravimetry (DTG) curves of four kinds of preprocessed SC are shown in Fig. 5. Thermal degradation took place in three stages for both the untreated and treated SC. From 100 to 200 °C, the slight decline in mass could be attributed to the inherent moisture in the fiber (Feng *et al.* 2020). The untreated SC showed the most significant weight loss at this stage, because of the lower moisture content of the dry-heat treated SC. The second stage was between 200 and 450 °C, at which point all the curves presented a rapid downward trend. In this phase, the mass loss was mainly caused by the degradation of the seed fibers. In the fiber, hemicellulose was the least thermally stable component, beginning to degrade at 230 °C and completely degrading at approximately 300 °C. The degradation of lignin started at 300 °C and was complete at 450 °C. The initial temperature of cellulose degradation was 275 °C and the final temperature was 550 °C (Hernández-Montoya *et al.* 2009; Oza *et al.* 2014; Feng *et al.* 2020). The final stage started at 500 °C, at which point the remaining non-pyrolytic components of the seed coat continued to decompose, the pyrolytic residues were further carbonized, and the mass loss tended to gradually stabilize.

There is a high temperature pretreatment process in the production process of SC particle filling material, and SC is usually sterilized by ultra-high temperature process to protect the user when used as a filler material for artificial turf. Therefore, thermal stability was a major condition to ensure that its durability was not affected before and after pretreatment (Xia *et al.* 2018). As seen in Fig. 5, the initial thermal degradation temperature of untreated SC (197 °C) was significantly lower than that of pretreated SC, and pyrolysis residue was the lowest (26.1%). Therefore, the thermal stability of SC improved after the pretreatment. The thermal stability of SC treated with a combination of polishing and dry heat could be regarded as the best, because of their superior thermal degradation temperature (214 °C) and pyrolysis residue (31.9%). This result might be due to the removal of ash and rough parts of the seed coat by polishing. Furthermore, part of the seed coat was shed due to dehydration after high-temperature dry heat treatment. For the comprehensively treated SC, it showed higher thermal stability because only the structurally stable parts of the seed husk and embryo remained (Hernández-Montoya *et al.* 2009; Oza *et al.* 2014).

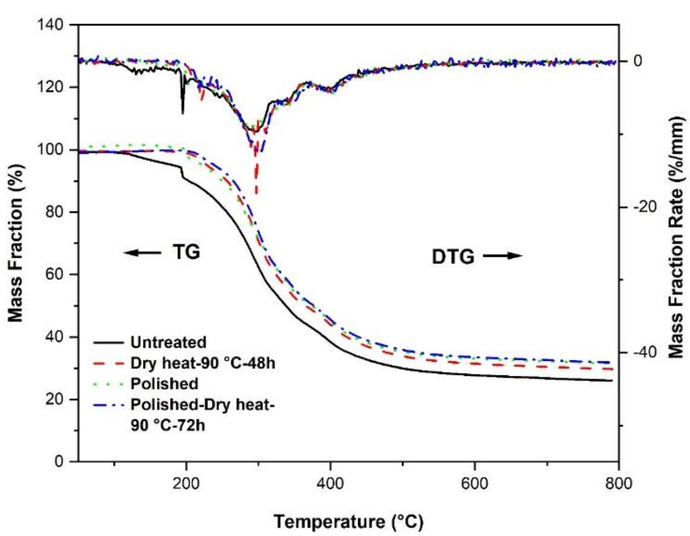

Fig. 5. The TG/DTG curves of four kinds of the preprocessed SC

Kinetic Analysis

The linear fit of log(β) of with 1/T was carried out based on Eq. 1 and experimental data. The activation energy was calculated by the linear slope, as seen in Fig. 6. The calculation results are shown in Table 3. The activation energy of the untreated and polishing SC was 101.4 kJ/mol and 147 kJ/mol, respectively. The activation energy of the untreated and polishing SC increased to 148.7 kJ/mol and 216.6 kJ/mol, respectively, after the dry heat treatment. The activation energy of SC significantly increased after all three pretreatments, among which the combination of polishing and dry heat had the largest impact on the activation energy.

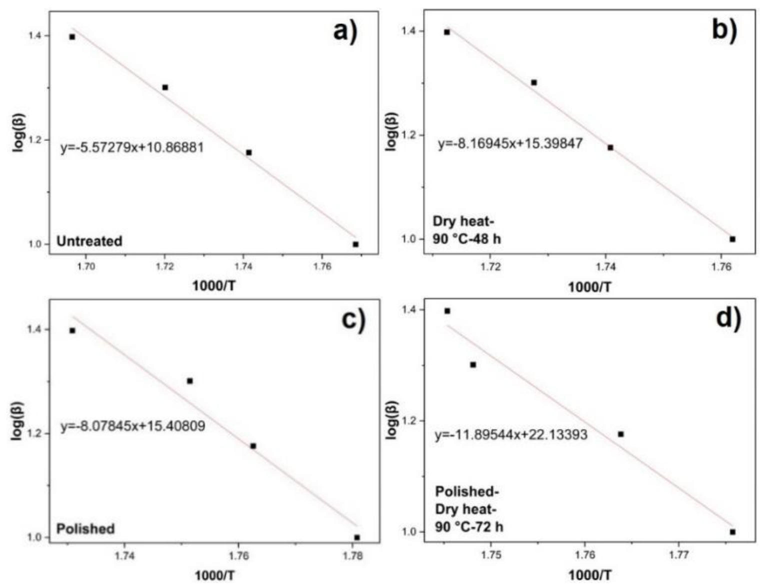

Fig. 6. Linear fitting of log(β) with 1/T for the SC that were a) untreated, b) dry heat treated at 90 °C for 48 h, c) polished, and d) polished and dry heat treated at 90 °C for 72 h

Table 3. Thermal Decomposition Kinetic Parameters of the SC

Treatment	β (K/min)	T_p (K)	E (kJ/mol)	R^2
Untreated	10	292.3	101.45	0.98958
	15	301.1		
	20	308.2		
	25	316.3		
Dry heat -90 °C and 48 h	10	294.4	148.72	0.99571
	15	301.3		
	20	305.7		
	25	310.8		
Polished	10	288.4	147.07	0.96538
	15	294.2		
	20	297.8		
	25	304.6		
Polished- Dry heat- 90 °C and 72 h	10	290.0	216.55	0.96814
	15	293.8		
	20	298.9		
	25	299.8		

A higher activation energy of SC meant that more energy was required to start a chemical reaction, which signified better thermal stability (Wang *et al.* 2017). Therefore, a higher activation energy of seeds with different treatments will yield superior thermal stability. This was consistent with the results of the thermal analysis. Furthermore, the chemical reaction rate of seed germination such as hydrolysis of carbohydrate and fat and transformation of protein was related closely to the value of the activation energy (Jiao *et al.* 2014; Edreis and Yao 2016; Wang *et al.* 2017; Xu *et al*. 2020). The increase in activation energy after pretreatment symbolized that that the energy required to perform the above reaction increased while the reaction rate decreased, which resulted in a lower germination rate of SC. And among the pretreatments, outstanding results were obtained with the combination treatment of polishing and dry heat taking the lead in the increase of the activation energy of SC, which was 113% higher than that of the untreated SC. This may be the main reason why the SC barely germinate after combined treatment.

Microscopic Morphology Analysis

Figure 7 shows the surface microstructure of four kinds of SC. The surface morphology of SC varied from smooth, slightly crackled, to crackled (Lubna *et al.* 2019). The untreated seeds had slight cracks on the surface, which were few and shallow (Fig. 7a). After dry heat treatment (90 °C, 48 h), the surface of SC was smooth, and a small amount of seed coat residue appeared (Fig. 7b). Presumably, the slightly crackled outer epidermis of SC would be brittle and fall away in a high-temperature dry environment, exposing a smoother layer of the seed coat.

Mechanical scarification appeared on the surface of the polished SC and the cracks on that deepened (Fig. 7c). It can be seen from Fig. 7c that SC had a thick and opaque texture (Hadidchi *et al.* 2020). After the comprehensive treatment of polishing and dry heat (90 °C, 72 h), the previous crack expanded into a depression, and the subcutaneous tissue was exposed (Fig. 7d). The seed activity decreased, and the structure of the seed coat was severely damaged under this treatment condition. For the reason that the hard and impermeable seed coat prevents SC from absorbing water and expanding, the water absorption of SC will presumably be enhanced subject to the cracks over the seed coat (Pereira *et al.* 2010). And the breakage of the seed coat renders the SC texture soft, where its resilience as a filling granular will deteriorate.

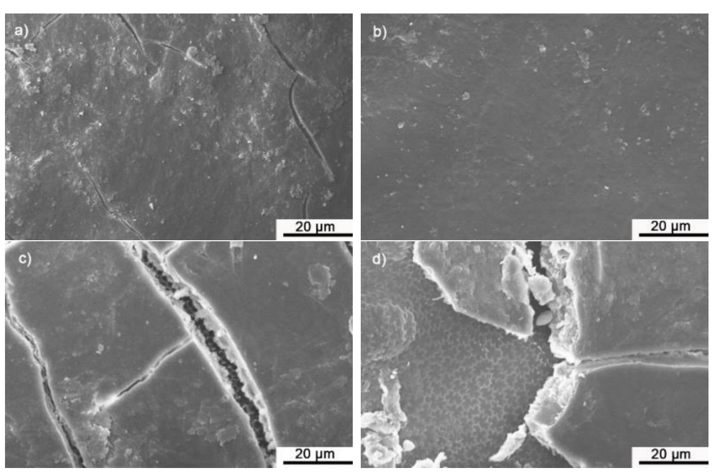

Fig. 7. Surface microstructure images of the a) untreated, b) dry heat treated, c) polished, and d) combined treated SC

Comparison of the Resilience of SC as a Filler Material

The resilience of the filler material has been defined as the capacity to absorb energy in the linear elastic range of mechanical deformation when it is impacted (Arthur *et al.* 2012). Resilience is a crucial factor to determine its cushioning on the human body when SC is used as a filling material for artificial turf (Ge *et al.* 2000). The surface structure of SC was damaged in varying degrees by dry heat treatment and polishing treatment. Although the germination problem was resolved, the performance of SC may be degraded. Therefore, the resilience of three kinds of pretreated SC was tested and compared with that of untreated SC.

The test results of the resilience testing are shown in Fig. 8. It was found that the resilience rate of the SC after polishing was the lowest (3.60%) among the three treatments and there was little difference with that of untreated SC (3.73%). Furthermore, the resilience rate of the SC after the dry heat treatment and comprehensive treatment increased to 4.23%, and 4.13%, respectively. Comparatively, the pretreatment did not weaken the resilience of SC as filling material, but rather it had a positive effect on the resilience. The reason for this result might be that the seed coat surface appeared pitted after the pretreatment, so the filling SC had a more compact stacking structure. In addition, the roughness of the seed coat increased after the dry heat treatment, which led to the poor overall fluidity of the filling material and a higher resilience rate (Li *et al.* 2015).

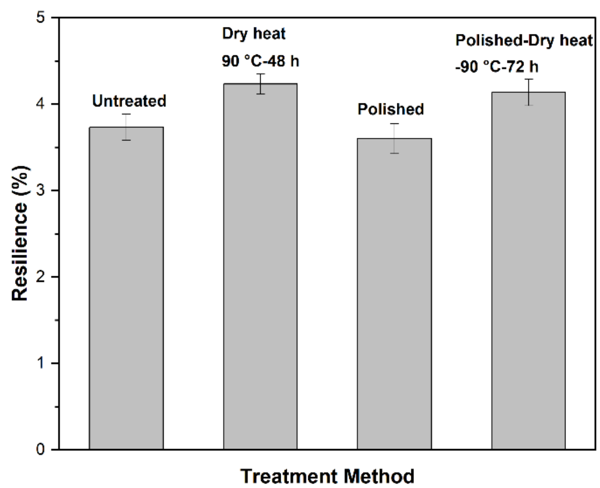

Fig. 8. The resilience of the SC as a filler material

Comparison of the Water Absorption for the Different Pretreated SC

Water inhibition, oxygen uptake, and increase in seed mass are the basis of seed germination (Wang *et al.* 2020). SC is often used outdoors as artificial turf filling granules, and the influence of rainy weather on its performance is non-negligible. As a consequence, the water absorption was also an important characteristic concerning the durability of the filler material, which might be impaired by dehydration and mechanical damage. The different pretreated SC were soaked in warm water (25 °C) for 24 h to observe the variation of the water absorption. As can be seen from Fig. 9, the water absorption of the pretreated SC was much higher than that of the control. The water absorption of the untreated SC was 54.3%, while that of the pretreated SC exceeded 100%. Most obviously, the water absorption of SC after the combined treatment reached 169%. The untreated SC had a hard and impermeable seed coat which normally prevented water imbibition. Therefore, the increase in water absorption could be attributed to the destruction of this hard seed coat (Wong *et al.* 2019; Wang *et al*. 2020). The above analysis was consistent with the observation of the microstructure.

Seeds that absorb larger amounts of water are less tolerant to the deterioration of the culture environment caused by temperature and humidity fluctuations that occurred under uncontrolled conditions, which negatively affects the physiological activity of seed germination (Abati *et al.* 2022). The resultant increased oxygen consumption from the higher water absorption, with a rise by 210% compared to untreated SC, can be inferred that the germination courses of the SC after the combined treatment require more oxygen, which would delay or prohibit germination (Dahal *et al.* 1996; Veselova and Veselovsky 2006; Liu et al. 2016). According to the analysis results of the activation energy, it can be implied that the increased activation energy and water absorption result in almost no germination of the SC after the combined treatment.

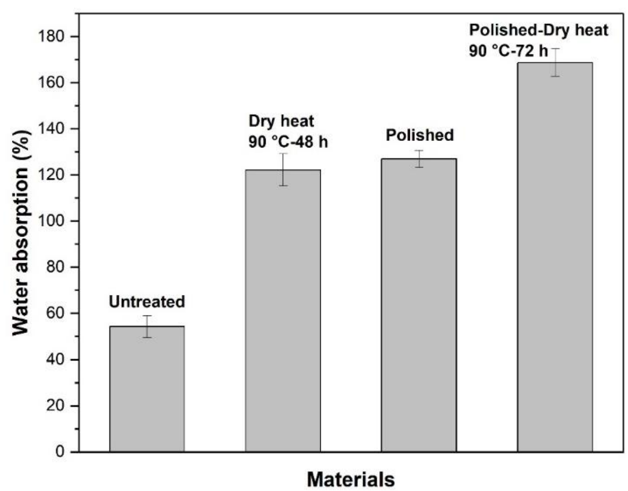

Fig. 9. The water absorption of the SC in 24 h

CONCLUSIONS

1. The combination of polishing and dry heat (90 °C, 72 h) was the most effective treatment to inhibit the germination of *Semen cassiae* seeds (SC), and the germination rate was reduced to 0%. It could be seen from the surface microstructure of SC that the seed coat was damaged and indented after combined treatment; however, its performance was not worse, the thermal stability of SC improved, and the resilience as an artificial turf filling particle increased from 3.73% to 4.13%.
2. SC had the highest activation energy and water absorption after combined treatment, at 217 kJ/mol and 169%, respectively, which increased the energy required for chemical reactions and respiratory oxygen consumption during the course of germination that would be further hindered.
3. The SC after polishing and dry heat treatment could be considered as environment-friendly and sustainable granular filling materials for artificial turf.

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