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Research Article

Polyester/Kota Stone Dust Composite: A Comprehensive Investigation of Mechanical and Sliding Wear Properties

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ABSTRACT

In the Kota stone industries, a huge quantity of unwanted waste is generated during the process of manufacturing stone. This waste is generally in the form of minute particles that are dumped on nearby land and carried away by water and air causing pollution in air, water, and soil. The most appropriate way is to convert this waste into a useful product. The present work comprises of fabrication and characterization of a new class of polymer composite with polyester as the base matrix and micro-sized Kota stone dust as filler material. A series of composites are fabricated at varied contents of micro-sized particulates using the open molding method. The properties explored are mechanical properties and sliding wear properties as a function of filler loading. It is noticed from the experimentation that the inclusion of filler material improves the different mechanical properties of the composites. The hardness of the composite along with the compressive strength improve by 25.37 % and 13.78 % respectively when 40 wt. % of the Kota stone dust is added to the polyester resin. The maximum hardness obtained is 84.2 Shore-D number and the maximum compressive strength of 109.2 MPa is registered. For a filler loading of 30 wt. %, the highest value of tensile strength is 66.2 MPa and that of flexural strength is 73.2 MPa is registered. The sliding wear tests are conducted as per Taguchi's design of experiment following the L_{25} orthogonal array. It is observed that the Kota stone dust loading is the utmost significant factor, whereas normal load is the least significant factor that administrates the sliding wear rate. The wear loss mechanism is observed under a scanning electron microscope by studying the eroded surface.

1. Introduction

Kota City is facing a major problem in disposing of the leftover produced from the Kota stone industry which is in the form of slurry powder. This slurry powder is obtained in different stages of processing of Kota stone like cutting, grinding, and polishing operations. In all, around 30-40 % part is wasted in the form of powder and slurry. The waste is deposited in the area which covers a major part of Kota city and some parts of the Jhalawar district. It is estimated that every year, the generation of slurry reaches a value of 3.25-3.5 lakh metric tons and the same is increasing exponentially. This slurry is disposed of in the nearby land and because of this around 5-10 hectares of land are wasted which can otherwise be utilized for some useful work [1]. Apart from that, it causes environmental and health problems in the locality. The problem arises mainly because of the very minute size of the waste as it is easily carried away by water and air and can also get into

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the underground water resulting in all types of pollution [2]. Hence, the consumption of this waste material for the production of some useful things should be on high priority. The work so far testified on the utilization of this waste is mainly on the manufacturing of Portland cement, bricks, ceramic tiles, and road construction. Apart from that, scientists also utilized this waste as a filler or soothing and pozzolanic material. However, studies concerning the usage of this waste in polymeric composites are very limited. However, a similar category of materials i.e. marble dust and granite dust has been extensively used as filler material for developing a polymeric composite.

A few examples of the utilization of marble dust and granite dust for the polymeric resin are discussed here. Awad and Abdellatif [3] evaluated the mechanical and thermal properties of the marble dust-reinforced LDPE composite. They reported that the addition of marble dust in the LDPE polymer improves the mechanical properties and thermal stability of the composites under investigation. Nayak and Satapathy [4] found that the mass loss due to wear of the composite reduces as the loading of marble micro-particulates in the polyester resin increases. Khan et al. [5] found improvement in mechanical properties and thermal conductivity of the LDPE polymer when the matrix material is added with marble dust and the increment is a function of a filler loading. Lendavai et al. [6] performed a complete investigation on poly(lactic acid) (PLA) filled marble dust composites. In the study, they found that when the content of marble dust increases in the PLA matrix, the flexural strength and impact strength also increase for a limited content of 10 wt. %. Further, they reported that the mass loss due to wear also reduces appreciably when the content of filler in the matrix increases. Singh et al. [7] also reported a similar behavior in their work.

Few works are reported on the size of the particles on different properties of the composites. Awad et al. [8] reported that the smaller size marble dust particles when reinforced in polypropylene polymer give better properties in terms of hardness and flexural strength. Nayak and Satapathy [9] reported that tensile and flexural strength are better with smaller size particles, whereas hardness and compressive strength are better with larger size particles when they work on polyester/marble dust composites. Apart from the size of the filler, the properties of the composites are further improved by the surface modification of marble dust particulates. Fiore et al. [10] treated the marble dust micro-particulates with plasma and studied the influence of this treatment on the properties of their epoxy-based composites. from the analysis, they found improvement in various properties of the polymer composites under study.

Garijipati and Malpapuram et al. [11] used micro-sized granite stone dust in poly benzoxazine polymer and observed that the inclusion of granite dust gainfully improves the hardness and the thermal stability of the composites. Mathavan and Patnaik [12] also reported improvement in the hardness of the polyester resin when micro-sized granite particulates were added. They also found a reduction in mass loss due to erosion wear when fillers are added to the polymer. The combination of granite dust and marble dust in a hybrid combination is also found to have a positive impact on the different properties of the polymer composite. Awad et al. [13-14] found that the hybrid combination of fillers enhances the flexural strength till the filler content of 50 wt. % whereas, the compressive strength and hardness increase with hybrid fillers for the entire filler content. Under the investigation of thermal properties, they reported improvement in thermal stability and decrement in the CTE of the composites, whereas melting temperature remains unaffected by such a combination. Against that, Karbous et al. [15] reported a decrement in flexural strength as the concentration of hybrid filler increases in a polystyrene matrix. In recent work, Maluga et al. [16] found that the hybrid combination of fillers successfully lowers the mass loss quantity under sliding wear to a great extent.

Apart from particulate filler composites, other categories of composite material are also in great demand which comprises various types of reinforcement material. The major work is going on the composites comprised of natural fibers as a reinforcement material which can either be single fibre composites or hybrid fibre composites where the natural fibers are used in combination with synthetic fibers [17-19]. Such fibre reinforcement composites generally enhance the mechanical properties of the matrix material. These composites belong to the category of green composites. Another category of composites that are in great demand is laminated composites [20-23] or composites prepared with nanotubes as a reinforcement material [24, 25]. It is observed by different researchers that such composites are useful where strength and intrinsic properties are of importance. Alimerzaei et al. [26] performed a nonlinear static vibration analysis of viscoelastic micro-composite beam-reinforced boron nitride nanotube (BNNT) using finite element method (FEM) and reported to achieve fruitful results.

Kota stone also belongs to the family of stones with which marble and granite dust belong and because of that, there is not a major difference in the composition as well. So, it has been said that, like marble and granite dust, Kota stone dust can also be implemented as a reinforcing material for the development of polymeric composite. Despite that, very little work has been reported in the past related to that. Rajput et al. [27, 28] are the ones who explored Kota stone dust for the development of polymer composites and gave useful findings. The different mechanical and tribological properties are found to improve in their work when Kota stone dust is embedded in the epoxy matrix. The work performed on Kota stone dust is only with an epoxy matrix. Given this, in the current work, a novel composite has been prepared with a combination of polyester resin and micro-sized Kota stone dust (KSD). The samples are fabricated at varied loading of filler and the effect of filler content on different physical, mechanical, and tribological properties is evaluated.

2. Materials Used

The matrix material used is an unsaturated isophthalic polyester resin and the same is supplied by Carbon black composites, Mumbai India. Methyl Ethyl Ketone Peroxide (MEKP) catalyst and cobalt accelerator are used with unsaturated polyester for curing. The reason for selecting the polyester as base matrix is because of its reasonably good mechanical properties over other polymers of similar category. Kota City of Rajasthan, India facing a foremost problem with disposing of the slurry that originated from the Kota stone industry. So, the dust obtained after drying this slurry is implemented as filler for the fabrication of polymeric composites.

3. Composite Fabrication and Characterization

A simple hand lay-up technique is used for fabricating the composites [29]. The following steps are followed for fabricating the composites:

a) The room-temperature curing polyester resin is added with the given percentage of micro-particulates and mixed well through hand-stirring for two minutes to ensure the homogeneous distribution of filler in the matrix body.

b) This is followed by the addition of 1 wt. % cobalt accelerator which was mixed properly. It is confirmed by the uniform change in color of the combination. The mixing time is kept at around 60 seconds.

c) Later, 1 wt. % of MEKP is added which was again hand-stirred for another 60 seconds to ensure the proper mixing of all the substances.

d) The uniformly prepared dough is then poured into the respective mold and kept there for 4 hours (curing time) so that it will get cured.

e) After 4 hours, the composites are carefully taken out from the mold and kept at room temperature for around 48 hours before being tested.

Table 1 shows the composition of samples prepared in the present work. Figure 1 shows the actual pictures of the samples prepared for conducting the various tests. Five samples of each composition are prepared and tested and the average of values obtained is presented in the result part.

A water immersion technique is used to experimentally determine the composite density as per ASTM D 792-91 standard. For comparison purposes, the density is also evaluated theoretically using an established rule of the mixture model. Using the two densities, air trapped is evaluated by measuring the percentage deviation between the two [30].

Table 1. Samples prepared in the present investigation

S. No.	Samples	Composition
1	Set A0	Neat Polyester
2	Set A1	Polyester + 5 wt. % KSD
3	Set A2	Polyester + 10 wt. % KSD
4	Set A3	Polyester + 15 wt. % KSD
5	Set A4	Polyester + 20 wt. % KSD
6	Set A5	Polyester + 25 wt. % KSD
7	Set A6	Polyester + 30 wt. % KSD
8	Set A7	Polyester + 35 wt. % KSD
9	Set A8	Polyester + 40 wt. % KSD

Fig. 1. Pictures of the fabricated samples for conducting various tests.

The tensile tests, flexural tests, and compressive tests are performed using a computerized Instron 3382 Universal testing machine following ASTM D638, ASTM D2344-84, and ASTM D695 standards respectively. The hardness is measured using the PosiTector SHD Shore hardness Durometer as per ASTM-2240 standard. A DUCOM pin-on-disc tribometer is used for conducting the sliding wear test as per ASTM G-99. The experiments are designed using Taguchi's design of experiment method. The control factors selected are sliding velocity, normal load, sliding distance, and filler loading [31]. The tests are conducted as per the L_{25} orthogonal array.

Table 2 shows the various control factors and levels used for experimentation. The specific wear rate is calculated from the measurement of loss of mass (Δm) in grams along with the density of material (ρ) in g/cm³, sliding distance (L) in m, and applied normal load (N) in Newton using equation 1 [28,29]:

$$W_s = \frac{\Delta m}{(\rho \times L \times N)} \text{ cm}^3/\text{N-m} \quad (1)$$

Table 2. Control factors and their values considered for the wear investigation

Control factors	Levels I	II	III	IV	V	Units
A (Sliding Velocity)	63	126	188	251	314	cm/sec
B (Normal Load)	5	10	15	20	25	N
C (Sliding Distance)	500	1000	1500	2000	2500	m
D (Kota stone dust Content)	0	10	20	30	40	wt. %

'Smaller is the better' characteristic is used to determine the S/N ratio using equation 2 as the minimum wear rate is the requirement [32, 33]:

$$\frac{S}{N} = -10 \, log \frac{1}{n} \sum y^2 \quad (2)$$

where n is the number of observations and y is the observed data.

4. Results and Discussion

4.1. Physical Properties

Figure 2 shows the density of the composite evaluated by two different methods along with the contents of the void present in the composites. The addition of Kota stone dust in the polyester matrix increases the density of the composites and the increment in density is linearly with filler loading.

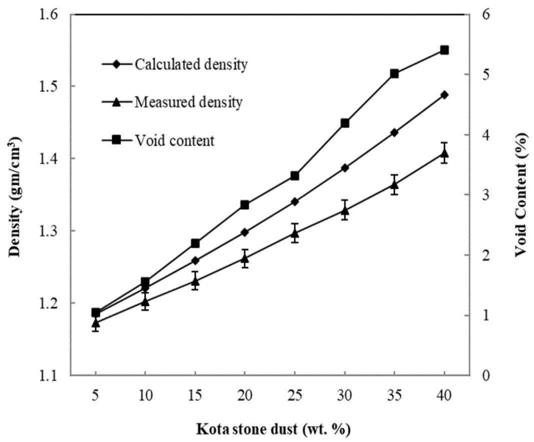


Fig. 2. Density of the polyester/KSD composites

The increasing trend in the density is observed for both experimental density and theoretical density. The said trend is obtained mainly because the intrinsic density of Kota stone dust is higher than the intrinsic density of the polyester resin. The measured density of neat polyester is 1.152 gm/cm³. With the inclusion of 40 wt. % Kota stone dust, the density increases and reaches a value of 1.408 gm/cm³. The increment in the measured density is 22.22 % with the inclusion of 40 wt. % filler. Further, it is seen that the calculated density is higher as compared to the experimental density. It is mainly due to the existence of air gaps in the actual samples and because of that, the overall measured density is less as the density obtained theoretically does not account for the presence of voids. Figure 2 also presents the content of voids in the composites. The curve shows that the content of voids is increasing with filler loading due to the increment in density difference obtained by two different methods. The maximum void content of 5.41 % is observed for the maximum loading of particulates. The presence of voids is not desirable as it deteriorates the different properties of the composites but is inevitable as the samples are prepared via an open molding method.

4.2. Mechanical Properties

The different mechanical properties under investigation are presented in Table 3. It can be seen from the table that the inclusion of waste particulates gainfully enhances the tensile strength and flexural strength but the enhancement is limited to 30 wt. % filler content.

Table 3. Mechanical properties of the composites under investigation

Set	Tensile strength (MPa)	Tensile modulus (MPa)	Flexural strength (MPa)	Flexural modulus (MPa)	Compre-sive strength (MPa)	Hardness (Shore D Number)
A0	46.5	1435	53.2	1930	87.1	74
A1	48.9	1621	56.1	2087	91.5	74.6
A2	51.2	1956	59.2	2213	95.4	75.1
A3	55.2	2187	63.4	2489	98.9	76.4
A4	59.5	2422	68.3	2673	101.3	78.2
A5	62.8	2745	71.4	2912	104.5	80.3
A6	66.2	2978	73.2	3120	107.8	81.8
A7	61.4	3123	69.8	3357	108.5	82.9
A8	56.2	3276	64.3	3539	109.2	84.2

With a filler loading of more than 30 wt. %, both the strength starts declining. The maximum tensile strength obtained is 66.2 MPa and the maximum flexural strength is 73.2 MPa. The strength decreases at high filler loading because of improper wetting of fillers along with their agglomeration with the matrix body. The same trend has been observed recently by Sawlani et al. [29] in their study of polyester/blast furnace slag composite.

Against that, the other mechanical properties continuously increase as the content of micro-particulates in the polyester matrix increases. The tensile modulus upsurges from 1435 MPa for unfilled polyester to 3276 MPa for polyester filled with 40 wt. % filler. Similarly, flexural modulus also increases from 1930 MPa (neat polyester) to 3539 MPa (Polyester + 40 wt. % filler) showing impressive improvement in the modulus values.

It is further observed that the strength under compressive loading situations also increases as the content of micro-particulates increases. The maximum compressive strength of 109.2 MPa is observed showing an increment of 25.3 % for 40 wt. % filler loading. The hardness increases from 74 to 84.2 when the content of filler increases to 40 wt. % showing an increment of 13.7 %. The improvement in both properties is mainly because the filler imparts a strengthening effect in the polyester matrix. The trend obtained is in line with the work reported by Lohiya et al. [32] where they study the properties of epoxy/LD slag composites. Also, Rajput et al. [28] reported a similar trend when they used Kota stone dust with an epoxy matrix.

4.3. Sliding Wear Behaviour

Loss in the mass of the composite during sliding wear teat is the difference between the initial mass of the pin before the test and the final mass of the pin after the test. The loss in mass is then converted to volume loss of the material using equation 1. This volume loss calculated is called the specific wear rate which is later transformed as signal-to-noise ratio using equation 2. Both the calculated values for each conducted test are presented in Table 4.

Figure 3 shows the influence of the four parameters i.e. sliding velocity, normal load, sliding distance, and filler loading on the output of the experiment. The figure shows that the change in the sliding velocity (A) and the filler content (D) affect the specific wear rate of the composite significantly, whereas, variation in the other two parameters is not able to affect much the output. Similar results were obtained by Purohit and Satapathy [34] when they evaluated the sliding wear behavior of epoxy filled with waste obtained from the steel industries.

Table 4. Dry sliding wear test results with the corresponding S/N ratios

Test Run	A Sliding velocity (cm/s)	B (Normal Load) (N)	C (Sliding distance) (m)	D (KSD content) (wt. %)	Specific Wear Rate (10^{-5} mm^3/N-m)	Signal-to-Noise Ratio (dB)
1	63	5	500	0	2.788	-8.9059
2	63	10	1000	10	2.501	-7.9623
3	63	15	1500	20	2.049	-6.2308
4	63	20	2000	30	1.864	-5.4089
5	63	25	2500	40	1.748	-4.8508
6	126	5	1000	20	2.229	-6.9622
7	126	10	1500	30	2.158	-6.681
8	126	15	2000	40	1.848	-5.334
9	126	20	2500	0	2.912	-9.2838
10	126	25	500	10	2.618	-8.3594
11	188	5	1500	40	2.041	-6.1969
12	188	10	2000	0	2.999	-9.5395
13	188	15	2500	10	2.694	-8.608
14	188	20	500	20	2.368	-7.4876
15	188	25	1000	30	2.294	-7.2119
16	251	5	2000	10	2.791	-8.9152
17	251	10	2500	20	2.492	-7.931
18	251	15	500	30	2.399	-7.6006
19	251	20	1000	40	2.162	-6.6971
20	251	25	1500	0	3.188	-10.0704
21	314	5	2500	30	2.572	-8.2054
22	314	10	500	40	2.293	-7.2081
23	314	15	1000	0	3.402	-10.6347
24	314	20	1500	10	3.015	-9.5857
25	314	25	2000	20	2.704	-8.6401

It is said because the first graph which is for sliding velocity and the last graph which is for filler loading show a noticeable variation in the mean S/N ratio for different values of sliding velocity and filler loading, whereas, the second graph which is applied load and the third graph which is sliding distance is having almost constant mean S/N ratio value for different values of sliding distance and applied. The more the variation in the S/N ratio with the change in the parameter, the more the influence of that parameter on the output.

Table 5 presents the response table from which the conclusion is derived that among all the factors, the loading of Kota stone dust is of the highest significance, whereas applied load has the slightest significance in lessening the mass loss of the composite. The said statement is concluded based on the delta value obtained through Minitab software.

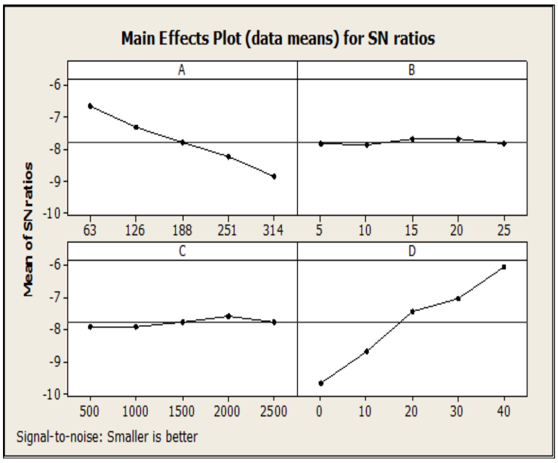


Fig. 3. Effect of control factors on the sliding wear rate of composites

From Figure 3 one more thing is derived the minimum wear is obtained for the combination of A1, B3, C4, and D5. This is concluded based on the peak obtained for every parameter on each graph of Figure 2.

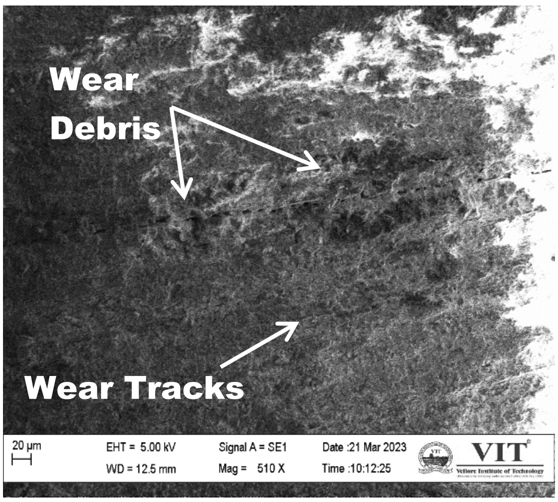
Table 5. Response table for a signal-to-noise ratio: smaller is better

Level	A	B	C	D
1	-6.672	-7.837	-7.912	-9.687
2	-7.324	-7.864	-7.894	-8.686
3	-7.809	-7.682	-7.753	-7.45
4	-8.243	-7.693	-7.568	-7.022
5	-8.855	-7.827	-7.776	-6.057
Delta	2.183	0.183	0.345	3.629
Rank	2	4	3	1

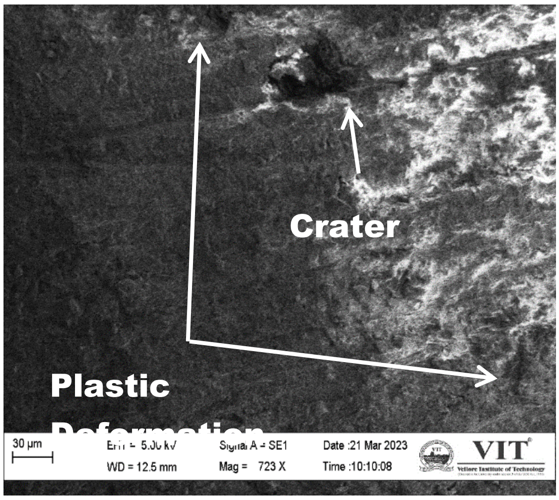
The peak for various parameters is 63 cm/s (sliding velocity), 15 N (applied load), 2000 (sliding distance), and 40 wt. % (filler content). Therefore, the above combinations of factors are confirmed to obtain the least specific wear rate.

4.4. SEM Analysis

The deteriorated surface of the prepared samples post-wear test is detected under SEM and their micrographs are studied to understand the prime cause of the material loss. The micrographs taken are presented in the figure 4. From the micrographs, it is clear that the surface of the composites is damaged by the pin during the sliding wear test. From the micrographs, it is also observed that the mass loss is less in the region where filler content is more. A crater is visible in the figure which is formed due removal of the matrix layer. A crater is a hole small or large hole developed on the surface of the body due to the removal of the material.



(a)



(b)

Fig. 4. SEM micrographs of composites' worn surfaces under various test conditions (a) Showing wear debris and wear track, (b) Showing crater and plastic deformation

The deformed layer of the matrix is removed as it forms a lump which was, they're at the top. This is removed by the transverse component of the applied load due to higher sliding velocity. However, the wear debris is much less on the deteriorated surface. This signifies that because of the sliding velocity, the initial debris is removed.

From the micrographs, it is clear that the loss due to wear is less, this is mainly because the Kota stone dust particles are hard. The hard nature of the filler material along with the good adhesion between the two phases results in less wear of the composite material under investigation. It is also visible that the tracks formed are discontinuous which means the wear is not uniform and the part where the pin comes in contact with the matrix encounters more wear whereas, when the pin comes in contact with the particles, the wear decreases significantly and results in the discontinuous track.

5. Conclusions

The physical, mechanical, and sliding wear properties of the polyester composites filled with varied content of Kota stone dust (5 wt. % - 40 wt. %) were determined experimentally. The inclusion of filler in the polyester resin increases the density and the air voids. While studying the mechanical properties, it is found that the compressive strength and hardness increase with filler loading. For a filler content of 40 wt. %, the compressive strength and hardness of the composite increase to 109.2 MPa and 84.2 Shore D number respectively which is 25.3 % and 13.7 % higher than the value of neat polyester. Against that, the strength under tensile and flexural loading does not show a continuously increasing trend with filler content, rather, for high filler content of above 30 wt. %, both the strength value starts decreasing. This confirms that excessive filler loading deteriorates the tensile and flexural strength of the composites. It is further found that with increased hardness, the Kota stone dust-filled polyester matrix shows a decreased specific wear rate. For specific wear, among the various factors, the Kota stone dust loading is of the highest significance whereas, the applied load is of the least significance. From the result, it is observed that the inclusion of micro-sized Kota stone dust gainfully improves the different mechanical and sliding wear properties of the material under investigation. With improved mechanical and tribological properties, it can be said that the presently fabricated composites can find their potential application in fields where specific mechanical properties are of more importance like in partition boards, doors and window panels, blades of wind turbines, etc.

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Conflicts of Interest

The author declares that there is no conflict of interest regarding the publication of this article.

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