Sustainable Lightweight Biochar-Based Composites with Electromagnetic Shielding Properties

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ABSTRACT: Global warming has prompted a search for new materials that capture and sink carbon dioxide (CO₂). Biochar is a derivative of biomass pyrolysis and a carbon sink mainly used to improve crop production. This work explores the underlying mechanism behind biochar’s electric conductivity using a wide range of feedstocks and its combination with a binder (gypsum). This gypsum–biochar composite exhibits decreased density and flexural moduli with increasing biochar content, particularly after 20% w/w. Gypsum–biochar drywall-like composite prototypes display increasing shielding efficiency mostly in the microwave range as a function of biochar content, differing from other conventional metal (copper) and synthetic carbon-based materials. This narrow range of electromagnetic interference (EMI) shielding is attributed to natural alignment (isotropy) of the carbon ultrastructure (e.g., lignin) induced by heat and intrinsic interconnectivity in addition to traditional phenomena such as dissipation of surface currents and polarization in the electric field. These biomass-derived products could be used as sustainable lightweight materials in a future bio-based economy.

INTRODUCTION

With the increasing global climate change due to CO₂ emissions, sustainable solutions involving materials with carbon-neutral or negative footprint urgently need to be developed and incorporated into mainstream manufacturing processes. Biochar is one such carbon-neutral or negative material that may play a role in manufacturing. Biochar is the solid product obtained from the pyrolysis of biomass (e.g., wood, agricultural wastes, municipal wastes), and is composed of stacked graphene sheets within turbostratic carbon crystallites with an amorphous component. If used for practically any application other than direct burning for energy, biochar is a carbon sink and classified as a material with negative global carbon footprint by virtue of its removal of carbon from global carbon cycles.

The predominant use of biochar until now is in agriculture as a soil amendment, where it has been used since pre-Columbian times in South American civilizations and other parts of the world. Intensive modern-day research has confirmed that biochar can improve soil fertility, detoxify soils, increase soil microbial diversity, and improve plant health. Biochar uses outside of the domains of energy and agriculture are limited, yet there are some efforts toward including biochar as a carbon-negative construction material. Its use in construction mainly focuses on improving the mechanical properties, performance, durability, and chemical stability of pavement-grade bitumen for road construction or for cement-based composites (for a review, see ref 21). Looking forward, the various physical and chemical properties of biochar, including its intimately entwined graphitic and amorphous carbon structures and electric conductivity properties, may hold novel applications in, for example, future lightweight sustainable building materials for construction with electromagnetic shielding properties.

In today’s modern technology-based lifestyle, it is impossible to avoid exposure to electromagnetic radiation. There are mainly three options to minimize exposure: time of exposure, distance from exposure, and shielding. Electromagnetic interference (EMI) shielding materials are based on two major mechanisms, reflection and absorption, as well as their combination. For electromagnetic reflection, the shielding effect is dictated by the interplay between electrical conductivity and magnetic permeability of the materials, and their interactions with electromagnetic radiation (EMR). Metals such as copper, gold, silver, aluminum, and nickel, or discontinuous carbon-based materials, such as carbon nanotubes or fibers, are commonly used. On the other hand, electromagnetic absorption is a secondary shielding effect created through the interaction between electric and/or magnetic dipoles of the material (e.g., BaTiO₃ or other...
materials with high dielectric constant) and EMR radiation (for review, see ref 26). Carbon-based materials displaying electromagnetic shielding effects have been largely explored for both reflection and absorption mechanisms, where the latter is achieved in combination with metal oxide nanoparticles.22,23,27−32

The electric conductivity of wood-based biochar has been explored as energy storage supercapacitors.33−36 The electric conductivity is thought to depend on the degree of carbonization and structural reorganization (e.g., graphitization and π stacking) of the biochar that occurs during heat treatment.33,37 The potential of biochar as an electromagnetic shielding material has been less investigated. Kushnood et al. described the use of carbon nano- and microparticles obtained by controlled pyrolysis of peanut and hazelnut shells (0.5% w/w) together with cement.31,32 These biochar–cement composite materials showed an electromagnetic shielding effect that was comparatively higher than composites based on equivalent mass fractions of carbon nanotubes. Although the concept is attractive, the use of carbon-sinking materials (biochar) composites with carbon-producing binding materials such as cement is a conflicting idea in terms of overall true sustainability. Thus, binders should have an environmental impact as low as possible. Gypsum (calcium sulfate dihydrate, CaSO₄·2H₂O) occurs abundantly in nature with virtually no toxicity for the environment and humans. It has been used as a building material for centuries with the largest expression during the Roman Empire, where it was used as a plaster. After mass structural devastation of World War II, drywalls became a highly demanded product up to present where is largely used in construction as drywalls or plaster.

The current work reports on the electric conductivity of biochar from different feedstocks and pyrolyzed under different conditions. Biochar from woodchips and eucalyptus is able to conduct electricity and shows the highest electric conductivity values. No direct correlation between the electric conductivity and degree of crystallinity was found. Based on elemental analysis, the electric conductivity is attenuated by the inorganic content that acts as insulator, and is directly dependent on the C/O ratio—the higher the C/O ratio, the higher the electric conductivity. Composites of woodchip biochar and gypsum show that increasing biochar content leads to lowering its density, with an abrupt decrease in flexural modulus for biochar content above 20% w/w. These gypsum–biochar composites display EMI shielding with increasing effectiveness with biochar concentration. There is also an increased shielding effect at frequencies above 4 GHz. This composite holds the potential to become the basis of a new generation of bio-based cost-effective construction materials such as bricks, interlocking bricks, or drywalls displaying not only negative carbon footprint using recycled organic material but also electromagnetic radiation shielding properties. In addition, it can be incorporated into mainstream manufacturing processes as new products.

■ RESULTS AND DISCUSSION
Electric conductivity was determined for biochar obtained from different feedstocks and pyrolyzed in a combinatorial way (Table S1 and Figure S1 in the Supporting Information). These feedstocks included treated sewage sludge, cattle manure, eucalyptus wood and woodchips, greenhouse pepper plant waste, olive pomace, date palm fronds, grain husks,
woodchips, and mixed wood wastes (Table S1). The classical four-point probe method of determining the electric conductivity was initially attempted. However, the pellets were too brittle when indenting with the probe. To overcome this limitation, a homemade setup was developed to measure the electric resistivity ($\rho$ in $\Omega \cdot m$) of powders. In the setup, the powder is compressed between two metal anvils with a constant known diameter. The thickness of each sample was measured separately and immediately after electric resistivity measurement using calipers. Among all biochar samples tested, the woodchip (highest treatment temperatures (HTT) 800 °C) and eucalyptus (A4.1, HTT 800 °C; treated with 20 mM H$_2$SO$_4$) biochars (Figure 1a) exhibited measurable electric conductivities of $6.18 \pm 1.5 \times 10^5$ and $3.25 \pm 0.5 \times 10^5$ S/m, respectively. A relatively simple electric circuit was built to demonstrate the electric conductivity. This electric circuit is regulated by a DC power supply of 5 V supporting a blue light-emitting diode (LED) bridged by a randomly picked biochar piece of a few centimeters in length. Figure 1b shows that when the circuit is closed, the blue LED turns on. In contrast, if there is no contact with the biochar piece (open circuit), the LED is turned off. X-ray fluorescence (XRF) analysis of all samples shows a correlation between electric conductivity and mineral content. Figure 1c shows that the biochar with a higher electric conductivity has less mineral content, suggesting that mineral content acts as an electric insulator. It was hypothesized that the degree of crystallinity derived from graphitization during the pyrolysis process would be responsible for the electric conductivity.33,37

The crystallinity index for the biochar obtained from various feedstocks was determined by calculating the ratio of the areas corresponding to crystal and amorphous regions of the X-ray diffractogram. Figure 1d shows a plot of crystallinity index for all biochar samples. Sample B9 (greenhouse waste consisting of pepper plant residues, HTT 600 °C) shows the highest crystalline index (82%) followed by samples O1 (treated sewage sludge, HTT 450 °C) and O2 (treated sewage sludge, HTT 600 °C) with 64 and 65%, respectively. Sample C6 (Olive pomace treated at 600 °C, HTT 600 °C) has a crystallinity index of 9.3%. Sample E1 (Grain husks, HTT 450 °C) and F1 (woodchips, HTT 450 °C) have crystallinity indexes of 8 and 61.3%, respectively. These results suggest that degree of crystallinity does not necessarily relate to the pyrolysis temperature but to the nature of the feedstock. Interestingly, Figure 2a,b shows that there is no correlation between the degree of crystallinity and electric conductivity. Sample A4.1 (eucalyptus) shows higher electric conductivity and crystallinity index of 18%. Sample B9 shows a higher crystallinity index (82%) but very low electric conductivity ($50.94 \pm 0.026$ S/m). This dataset suggests that degree of crystallinity (as determined by the crystallinity index) is not necessarily responsible for the electric conductivity found in biochar.37

Elemental composition in terms of C, O, H, and N content was measured from two samples that show the highest electric conductivity (A4.1 and Sp1) and four randomly chosen samples that show lower electric conductivity (C6, P1, D4, and B9). There is an overlap between the electric conductivity and C/O ratio. Figure 3a shows a plot of electric conductivity vs C/O ratio for two biochar samples that show the highest electric conductivity (A4.1 and Sp1) and four randomly chosen samples that show lower electric conductivity (C6, P1, D4, and B9). There is an overlap between the electric conductivity and C/O ratio. Figure 3b shows a plot of electric conductivity and C/O ratio vs different temperatures of pyrolysis of eucalyptus (HTT 350, 400, 500, 600, and 800 °C). As the temperature increases, the C/O ratio increases as well as the electric conductivity.

Figure 2. (a) Crystallinity index and electric conductivity vs different biochar samples pyrolyzed at different temperatures. (b) Crystallinity index vs electric conductivity. No correlation is found.

Figure 3. (a) Electric conductivity vs C/O ratio for two biochar samples that show the highest electric conductivity (A4.1 and Sp1) and four randomly chosen samples that show lower electric conductivity (C6, P1, D4, and B9). There is an overlap between the electric conductivity and C/O ratio. (b) Electric conductivity and C/O ratio vs different temperatures of pyrolysis of eucalyptus (HTT 350, 400, 500, 600, and 800 °C). As the temperature increases, the C/O ratio increases as well as the electric conductivity.
tested by pyrolyzing eucalyptus wood at different oxygen content, the higher the electric conductivity. This was shown in Supporting Information). The hypothesis is that the lower the C/O ratio by restoring the carbon sp² bonds network and thus its electronic properties. These results agree with the increase of electric conductivity and C/O ratio (Figure 3a) but not with C/H, H/C, C/N, N/C, N/H, and H/N ratios (Figure S2 in the Supporting Information). The hypothesis is that the lower the oxygen content, the higher the electric conductivity. This was confirmed by pyrolyzing eucalyptus wood at different temperatures (HTT 350, 400, 500, 600, and 800 °C) under the same experimental conditions. The samples were rinsed with H₂SO₄ (20 mM). The electric conductivity and elemental analysis measurements show that electric conductivity increases with C/O ratio as a function of increasing pyrolysis temperature (Figure 3b). These results agree with the increase of electric conductivity by reduction of graphene oxide, in which the removal of significant amounts of oxygen increases the C/O ratio by restoring the carbon sp² bonds network and thus its electronic properties. 36,39

Biochar-based composite materials were fabricated by combining different concentrations of woodchip biochar (0, 2, 5, 7, 10, 20, 30, 40, and 50% w/w) with gypsum (as a binder). After mixing, the samples were left to cure for 48 h at 60 °C. The effect of biochar filler on the density and mechanical properties of the composite was explored using a flexural strength three-point bending test. Figure 4a shows the composite beams with 0, 2, 10, and 20% w/w biochar. Their gray hue increases with biochar content. The density measurements of these bricks show average values of 1.08 ± 0.03, 0.99 ± 0.02, 0.98 ± 0.01, 0.97 ± 0.03, 0.93 ± 0.02, 0.82 ± 0.02, 0.61 ± 0.02, 0.49 ± 0.03, and 0.46 ± 0.03 g/cm³ for biochar contents of 0, 2, 5, 7, 10, 20, 30, 40, and 50% w/w, respectively (Figure 4b). The higher the biochar content, the lower the composite’s density. However, when considering an application, structural stability plays an important role. Thus, flexural moduli of these biochar composites were determined using a three-point bending test on rectangular beams (100 × 16 × 4 cm³) of gypsum-biochar composites with the same biochar content as for the density measurements. Figure 4c shows average flexural modulus values of 709 ± 12, 584 ± 12, 596 ± 17, 425 ± 18, 457 ± 21, 119 ± 25, 51 ± 18, and 48 ± 19 MPa for biochar contents of 0, 2, 5, 7, 10, 20, 30, 40, and 50% w/w, respectively. These results show that increasing the biochar content in the composite decreases the flexural modulus, with an abrupt drop in flexural modulus at 30% w/w (Figure 4c). Composites with mass fractions beyond 40% w/w are very crumbly. At mass fractions above 80% w/w biochar the composite is electrically conductive and could find applications as conductive paste that could be placed in continuous grooves to serve, for example, as conductive wires for temporary housing (Figure S3 in the Supporting Information).

The composite’s lightweight properties, the wide use of gypsum as construction material, and the fact that the biochar is intrinsically electrically conductive, prompted further exploration of this bio-based composite to act as Faraday cage to shield electromagnetic radiation in the format of a construction material prototype. For this purpose, drywall-like plates (600 × 300 × 10 mm³) were fabricated with 0, 5, 10, 20, 30, 40, and 50% w/w biochar content (Figure 4d). The plates containing 50% w/w of biochar did not have sufficient structural integrity to pursue further studies (Figure S4 in the Supporting Information). Current fabrication of drywalls include formulation with additional binders. This formulation could allow the composite to withstand higher content of biochar and, consequently, increase its mechanical properties. Shielding attenuation tests were carried out on drywall-like...
plates to assess the effectiveness of electromagnetic radiation shielding properties of the following composites: 0, 10, 20, and 40% w/w content of biochar. The tests were performed according to the standards ENS0147/MILSTD-285/NSA65-6 in the frequency range 800 MHz to 6 GHz under environmental controlled conditions and in an anechoic chamber. The plates were fixed in a metal frame with screws distancing 60 cm apart from the transmitting and receiving antennas (Figure S5 in the Supporting Information). Figure 4e shows the electromagnetic shielding effectiveness (dB)—here represented as the sum of all of the losses—plotted against the emitting frequency range of 800 MHz to 6 GHz. The gypsum alone is an electrical insulator and does not contribute to electromagnetic shielding (Figure 4e black line). In all three cases, there is an increase of the shielding effectiveness with the increase of the biochar content, very likely attributed to the proximity of the biochar particles. Interestingly, the shielding effectiveness of the drywall-like plates with 10, 20, and 40% w/w contents of biochar is significantly higher at frequencies beyond 4 GHz. At 1 GHz, shielding effectivenesses of the drywall-like plates with 10, 20, and 40% w/w contents of biochar are 3.0 ± 0.35, 5.5 ± 0.1, and 9.5 ± 5.6 dB, respectively. These values are significantly lower than 40 and 74 dB described for cement–carbon composite containing 1.5% w/w of discontinuous 0.1 μm diameter carbon filaments43 or a thermoplastic polymer matrix containing 19% w/w carbon filaments or graphene paper (110 dB), foams supplemented with carbon nanotubes (75 dB)42 (see ref 41 for a comparison table), respectively. In the microwave region, in particular at the frequency of 5 GHz, shielding effectivenesses of the drywall-like plates with 10, 20, and 40% w/w contents of biochar are 7.65 ± 1.6, 12.85 ± 0.1, and 13.6 ± 0.56, respectively. At the maximum frequency recorded (6 GHz), these values rise to 11.65 ± 1.6, 19.2 ± 5.7, and 19.25 ± 1.8, respectively. These frequencies fall in the range of future radio-based technologies such as the low- and middle-band 5G for cell phones with frequencies ranging from 3.5 to 4.2 GHz and from 4.4 to 4.9 GHz, respectively, a region where the biochar displays higher shielding effectiveness. Conventional metal-based EMI shielding materials display a relatively weak microwave absorption due to surface reflection.42 Carbon-based materials such as carbon nanotubes and graphene and polymer composites therefore have been explored as alternatives for EMI shielding applications mainly due to their high electric conductivity, high surface–area ratio, low density,43 and ability to reflect and absorb radiation44 also in the microwave region. Biochar differs from other conventional metal (copper) and synthetic carbon-based materials because it shows shielding effectiveness mainly in the microwave range. Although the mechanism is not yet fully understood, this observation can be attributed to natural alignment (isotropy) of the carbon ultrastructure (e.g., lignin) induced by heat and intrinsic interconnectivity in addition to traditional phenomena such as dissipation of surface currents and polarization in the electric field.44

## EXPERIMENTAL SECTION

### Biochar and Pyrolysis of Different Feedstocks. A tabulated list of feedstocks, pyrolysis conditions, and additional treatments when relevant is found in Table S1. Briefly, feedstocks included treated sewage sludge, eucalyptus wood, cattle manure, date palm fronds, greenhouse pepper plant wastes, olive pomace, grain husks, woodchips, and mixed wood waste. In all cases, the technology used was slow pyrolysis batch processing, at atmospheric pressure and with varying imposed highest treatment temperatures (HTT) as detailed in Table S1.

**X-ray Fluorescence (XRF).** Bulk chemical composition was obtained by energy-dispersive X-ray fluorescence (ED-XRF), using a Spectro-XEPOS instrument (SPECTRO Analytical Instruments GmbH, Germany) with a palladium (Pd) anode and a set of secondary targets. The samples were analyzed as powders under room temperature and atmospheric pressure. Approximately 3 g of each sample was homogenized and ground to a fine powder using an agate mortar and pestle. The ground samples were then placed in an XRF plastic cup with a 4 μm thick Prolene film (Chemplex Industries, Inc.) covering the window. Precision and accuracy were tested by repeated analyses of a standard reference material (GSS-1, Chinese geochemical standard reference materials for soils). The standard was measured in each of the sediment runs (12 samples in each run). Under these conditions, accuracy is generally better than 10% for elements present in the analyte at more than 5%. Three samples were randomly picked from four different batches, and the values of the triplicate were averaged. Statistical treatment was performed using Origin Pro v8.0725 (OriginLab Corporation, Northampton).

**X-ray Diffraction (XRD).** Diffraction measurements were carried out in reflection geometry using an Ultima III (Rigaku, Japan) diffractometer equipped with a sealed Cu anode X-ray tube operating at 40 kV and 40 mA. A bent graphite monochromator and a scintillation detector were aligned in the diffracted beam. θ/2θ scans were performed under specular conditions in the Bragg–Brentano mode with variable slits. The 2θ scanning range was 10–70° with step size and scan speed of 0.02° and 0.5°/min, respectively. Phase analysis was performed using the PDF-4+ 2020 database (ICDD) and Jade Pro software (Materials Data, Inc.). The crystallinity index for the biochar obtained from various feedstocks was determined by calculating the ratio of the areas corresponding to the crystal and amorphous regions of the X-ray diffractogram.

**Electric Conductivity Measurements.** Electric conductivity was measured using a homemade device (Figure S1 in the Supporting Information for schematics). Biochar samples were powdered using an agate mortar and pestle. The powder was placed between two stainless steel cylinders with an area of 2 × 10⁻⁴ m² surrounded by a polymeric (polyether ketone, PEEK) ring. The cylinders were connected to a power source with constant current and voltage (5.19758 V) and a

## CONCLUSIONS

Biochar is a product of biomass pyrolysis and an abundant resource with a potential use beyond agriculture. This work explores the electric conductivity of biochar from different feedstocks and its potential application as lightweight drywall-like plates that can shield electromagnetic radiation predominantly in the microwave region as opposed to conventional metal-based materials. The electromagnetic shielding efficiency is a function of the biochar content. Thermal insulation properties of gypsum–biochar are an avenue worth exploring in the future to reduce energy consumption for heating or cooling buildings. This report demonstrated that pyrolyzed biomass can be used toward a new generation of bio-based lightweight sustainable functional materials and bioeconomy with electromagnetic radiation shielding properties in the microwave region.
multimeter with ±0.01 mV precision. Given the low resistance of the biochar (~mΩ), an additional 215 Ω resistance is connected in series to the sample. By measuring the current in the 215 Ω resistance and the voltage drop across the biochar, a measurement of sample resistance is obtained. The sample resistance combined with its geometry gives us a conductivity measurement. The sample placed between the cylinders was pressed under a hydraulic pump so that the contact is maximized and the values stabilized (2 tons of pressure). The voltage was measured directly from the multimeter. Immediately after the measurements, the thickness of the sample was determined using calipers. The samples were measured in triplicate, and the values were averaged. Statistical treatment was performed using Origin Pro v8.0725 (OriginLab Corporation, Northampton).

**Gypsum–Woodchip Biochar Composites.** Gypsum was commercially available (Krone Modelgips, Hilliges Gipswerk GmbH & Co. KG, Germany). The composites were prepared manually by mixing gypsum powder with milled woodchip biochar in different proportions: 0, 2, 5, 7, 10, 20, 30, 40, and 50% w/w. To this powder mixture, distilled water was added in the following proportion: 7 mL of distilled water to 10 g of gypsum. The composites were mixed until homogeneous, immediately poured into molds, and agitated to release entrapped air bubbles. The molds were placed in an oven for 48 h at 60 °C. The samples were removed from the molds, allowed to cool down to room temperature, and kept in the room until further use.

**Determination of Density and Flexural Modulus of Beams Made from Gypsum–Woodchip Biochar Composites.** Density was determined by measuring the volume of gypsum–woodchip biochar composites with 0, 2, 5, 7, 10, 20, 30, 40, and 50% w/w biochar using a caliper with ±0.1 mm precision, and the weight was measured using a scale with ±1 g precision (MRC Lab, Israel). The measurements were done in triplicate, and the values were averaged. For the determination of flexural modulus, rectangular beams (100 × 16 × 4 cm³) were fabricated. A three-point bending test was carried out using a Zwick Roell 1446 Universal Testing Machine (Zwick Roell, Germany) equipped with a 200 N load cell under the following conditions: pre-load, 0.05 N; test speed, 1 mm/min; room temperature. The assays were performed in triplicate, and the flexural modulus was calculated for each sample. The values of the triplicate were averaged, and statistical treatment was performed using Origin Pro v8.0725 (OriginLab Corporation, Northampton).

**Electromagnetic Field (EMF) Shielding Measurement on Gypsum–Biochar Drywall-like Plates.** Plates of 600 × 300 × 10 mm³ of gypsum–woodchip biochar composites 0, 10, 20, and 40% w/w were prepared using a similar procedure to that for the beams. Immediately after homogenizing the gypsum–biochar mixture, it was cast into a homemade polylvinyl chloride (PVC) frame containing recycled cardboard 600 × 300 × 2.5 mm³ thick (HEYDA Graupappe, Germany) at the bottom. Another similar sheet of cardboard was added after the casting process. The plates were dried in an oven at 60 °C for 48 h. Electromagnetic field (EMF) shielding attenuation tests were carried out according to standards EN50147/MILSTD-285/NSA65-6 in the frequency range 800 MHz to 6 GHz at 21 °C, 51% relative humidity (RH), 1013 hPa air pressure, and in an anechoic chamber. The assays were measured in duplicate and averaged. Statistical treatment was performed using Origin Pro v8.0725 (OriginLab Corporation, Northampton).

**ASSOCIATED CONTENT**

**Supporting Information**

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsomega.0c04639.

Schematic representation of the homemade setup to measure electric conductivity of the biochar powders (Figure S1); C/H, C/N, and N/H ratios of two biochar samples (Figure S2); schematic representation of a potential application of biochar–gypsum (Figure S3); photographic image of the drywall plate with 50% w/w biochar (Figure S4); representative photographic images of the shielding attenuation test (Figure S5); and pyrolysis conditions of different feedstocks (Table S1) (PDF)

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Notes

The authors declare no competing financial interest.

**ACKNOWLEDGMENTS**

The authors thank funding grant PCI-MEC 80170111 from CONICYT (Chile) and a Visiting Faculty Program Fellowship from the Weizmann Institute of Science. This work was financially supported by the research grant from the Benoziyo Endowment Fund for the Advancement of Science, Estate of Raymond Lapon, Estate of Olga Klein Astrachan (Weizmann Endowment Fund for the Advancement of Science, Rehovot, Israel), Estate of Harriet Nahum, Dora Yoachimowicz Endowed Fund for Research, Minzer Family Fund Scholarship, Gideon J. Hamburger, and Estate of Robert Einzig.

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