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Microwave Technology in Freeze-Drying Process

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Abstract

Dehydrating is one of the most common processes in industry. This process is implemented by various techniques, such as freeze-drying. It is an energy-consuming process. Microwave sources are a good choice to supply the energy needed for this process. In reality, it is microwave-assisted freeze-drying. The microwave sources can be delivered around a few kilowatts. Electromagnetic energy is converted into thermal energy due to the interaction of electromagnetic fields and materials. In addition to providing energy, the microwave-assisted freeze-drying is time-saving. This method is fast due to penetrating electromagnetic fields in the material. It results in volumetrically heating instead of heating from the surface of the material in conventional methods. Usually, the frequency of electromagnetic fields is 2450 MHz, which is allocated by regulatory commissions in dielectric heating methods. In the following, the mechanism of this method is described. All relations governing the transfer of mass and heat are mentioned. How to transfer and dissipate energy is described. Dielectric properties of different materials are listed. The effective parameters in determining dielectric properties are discussed.

Keywords: freeze-drying, dielectric heating, microwave energy

1. Introduction

Population growth of human societies results in increasing the demand for requirements such as food, clothing, housing, etc. Meeting them requires new industrial methods instead of traditional ones. The production of foodstuff is included in this principle. Today, different processes are being done on the mineral, vegetable, and animal products. Some of them are pasteurization, sterilization, conservation, etc. Each of them is used for a specific purpose. During these processes, physical, chemical, and biological changes occur. They affect the quality of foodstuff (color, flavor, volume) [1].

Drying is the most common way to increase the life of food products to make them easier to maintain [2]. Meanwhile, microwave technology has achieved a significant position among other methods in food industry. Not only is this method used in food industry but also in pharmaceutical industry and medical sciences, for removing water from aqueous solutions and preserving the blood, bone, and skin [3–5].

In conventional method for drying foodstuff, it is heated, usually by flowing hot air, to evaporate its moisture. Also, the heating can be done by other methods from direct solar radiation to using microwave energy [1, 6]. In freeze-drying method, removing the moisture content of material is done by sublimation of water molecules with internal heating after freezing the material and creating a vacuum [7]. Compared with conventional methods, it causes small irreversible changes in food and thus keeps the quality of product at an excellent level [1, 2, 7]. Rehydration, color (browning), and volume (volume reduction and consequently shrinkage) are key parameters in determining the quality of foodstuff and are considered in [1]. Low temperature in this method helps to stop most biochemical reactions, and hence it is suitable for dehydrating heat-sensitive material like biological products [1–3]. However, this method is expensive [1, 7]. It is suitable for valuable foodstuffs like coffee [1]. Accordingly, researchers are trying to find the optimal method by a combination of different methods.

Ref. [1] is a valuable review on the studies, which are done about the quality of foodstuff from different drying methods, and collects and presents different graphs about these parameters. Author in [1] presents a chart which determines the contribution of energy consumed in different operations of freeze-drying process. Also, the cost breakdown for drying two samples (high- and low-value foods) is determined in [1].

Drying (or dehydrating) is removing moisture content from a material. This phenomenon, which required phase change in water content of material, requires a lot of energy [6].

In the traditional method, the needed energy must be transferred from dried layer (into frozen bulk), which has the low thermal conductivity. This means that it takes long time [6]. Microwave technology helps to transfer the needed energy in a form of electromagnetic wave into the frozen region, independent of thermal characteristics of dried layer. Then, the electromagnetic field is dissipated in frozen region and increases its temperature. Since the field is distributed in the frozen region, the dissipation occurs throughout frozen bulk. In fact, it creates internally volumetrically heating [2, 6, 7].

In [8], it is mentioned that the volume reductions (shrinkage) for strawberry dried by freeze-drying and air-drying methods are around 6.6 and 80%, respectively [1, 8].

Ref. [3] considered the conventional and microwave-assisted freeze-drying methods. It showed that the drying time is less than 20% for microwave-assisted freeze-drying method because of volumetrically heating in this method.

2. The freeze-drying process

In freeze-drying process, the material must be frozen in the first step. Then, it is followed up with creating vacuum and injecting energy by microwaves in the chamber or storage compartment which contains the frozen food material [2] (**Figure 1**).

Figure 1. Steps must be passed in microwave-assisted freeze-drying process.

Figure 2. Profile of different layers with typical variations of temperature in them for an infinitely long slab of material with thickness L (reproduced from [2]).

What happens during these stages? The heating of the frozen food by microwave energy causes the frozen bulk temperature to increase. With increasing bulk temperature, frozen molecules of water receive enough energy and transit from solid phase to gas phase (sublimation of frozen molecule of water). These molecules migrate (from frozen bulk) into vacuum region of chamber. In other words, the moisture is removed from frozen region, and food material is dried. This sublimation starts from the outer layer. Now, a new region forms in frozen bulk from interface of food material and vacuum and dried region (**Figure 2**). As the time proceeds, the interface between dried and frozen regions will retreat. Therefore, the frozen bulk of material is thinned, and the volume of dried section increases [2].

According to the above discussion, we are faced with three physical phenomena: producing thermal energy by dissipation of microwave energy (in frozen bulk), heat transfer (in frozen and dried regions of food material), and mass transfer which is related to the movement (flow) of water vapor in the system (in the dried region). The heat transfer in the freeze zone is done by conduction, while the heat is transferred in the dried region in conduction and convection.

3. Heat and mass transfer equations

As mentioned previously, with increasing the temperature of bulk, two heat transfers are carried out in the bulk. The heat transfer in the frozen region is conductive, while the heat transfer in dried region is a combination of conductive and convective. The heat transfer in frozen region obeys the following relation:

$$\rho_F C_{PF} \frac{\partial T_F}{\partial t} + \vec{\nabla} \cdot \left(-\vec{k}_F \cdot \vec{\nabla} T_F \right) = p_d \quad (1)$$

where T_F , ρ_F , C_{PF} , $\vec{k}_F$, and p_d are temperatures of frozen region, density of frozen material, heat capacity of the frozen material, the vector of thermal conductivity of frozen material, and density of dissipated microwave power, respectively. All of them are for frozen zone. The convective transfer in dried region is due to the flow of water vapor through this region. Hence, in dried region, the heat transfer obeys the following relation:

$$\rho_D C_{PD} \frac{\partial T_D}{\partial t} + \vec{\nabla} \cdot \left(-\vec{k}_D \cdot \vec{\nabla} T_D \right) = p_d - C\vec{W} \cdot \vec{\nabla} T_D \quad (2)$$

where C and $\vec{W}$ are concentration and the vector of mass flux, respectively. These two parameters are related to water vapor. All other parameters are the same ones in the previous formula but for dried zone. This relation shows our need to know the concentration of water vapor. The water vapor concentration obeys the mass transfer relation. Relation (3) specifies the variation of concentration (C) of water vapor in dried layer:

$$\vec{\nabla} \cdot \left(\vec{D} \cdot \vec{\nabla} C \right) = \sigma \frac{\partial C}{\partial t} \quad (3)$$

where $\vec{D}$ and σ are the vector of effective diffusivity and porosity of the dried material, respectively. It is possible to find the distribution of temperature by simultaneously solving these equations. Initial and boundary conditions must be considered to solve these equations. Also, the thermodynamic equilibrium, governed at the interface of frozen and dried region, determines the relationship between the concentration of water vapor and the temperature of frozen region [2, 3].

It is evident that density and porosity of food material, along with moisture and fat content, are the key factors in the determining the process.

4. The transient variation of temperature during freeze-drying

When an infinitely long slab of the proposed material is available, it can be assumed that all variations happen in direction orthogonal to the surface of slab (one-dimensional variation). The typical distribution of temperature in different layers (frozen, dried, and vacuum) and the concentration of water vapor in dried layer are shown in **Figure 2**. It is sufficient to show the variation only in just half of the structure because of symmetry.

where T_c , T_i , T_v , C_i , and C_v are the temperatures in the middle of frozen bulk, the temperature of interface (between frozen and dried regions), the temperature of vacuum, the concentration of water vapor in interface (between frozen and dried regions), and the concentration of water vapor in interface of vacuum and dried regions [2]. The sublimation will continue until the temperature of dried zone (in the interface) is kept under the melting point of frozen region [2]. The variation of temperature in dried region is a part of the parabolic. It is valid for frozen region, too.

For beef meat, cut into a slab, the temperature distribution is demonstrated for different times by [2] (**Figure 3**). Its thickness is 1 inch and is in the middle of chamber.

From **Figure 3**, the concentration of water vapor in dried region is decreased by getting away from the interface (between frozen and dried layers). The necessary energy for this system is provided by electromagnetic waves at 2450 MHz. In [2], it is assumed that the electric field is approximately uniform throughout the material and its intensity equals to 12.5 KV/m. The total pressure and partial pressure of water vapor in the vacuum region are 0.29 and 0.075 mmHg, respectively. The drying time for this meat is measured 6 hours when applied electric field is nearly 10 KV/m [9]. Generally, the higher the electric field intensity, the smaller the drying time (**Figure 4**).

In [2], it has been shown that the optimum operation of freeze-drying process is obtained near the corona and melting point. For a slice of meat in a rectangular cross section, the variation of temperature and concentration happened in two dimensions. **Figures 5** and **6** are shown in simulation and experimental results [7].

Figure 3. The temperature (left) and concentration (right) profiles for a slab of beef meat in different times (reproduced from [2]).

Figure 4. Dry time for a slab of beef meat (reproduced from [9]).

Figure 5. The temperature profile along X and Y axes for a bar of meat with square cross section (reproduced from [7]).

Figure 6. The time variation of moisture content for a bar of meat with square cross section (reproduced from [7]).

Figure 7. A setup used to microwave-assisted freeze-drying process (reproduced from [2]).

Figure 7 shows a setup used in [9] for freeze-drying process by injecting microwave power. A microwave oven supplies sufficient adjustable energy. A magnetron with the capability of delivering 1.2 KW adjustable power at 2450MHz is used in the oven (A). The proposed material (D) is located in the middle of microwave cavity (B), and they are surrounded by a vacuum chamber (C). This cavity has dimensions $39 \times 39 \times 51$ cm, all of its walls are made of perforated aluminum sheets to supply the needed electromagnetic boundary conditions and provide a path to free flow of water vapor simultaneously. To avoid harmful reflection from the cavity, a circulator is used just after the generator. To absorb the reflected power, it is necessary to match other ports of the circulator. A twist (J) is used to change the polarization of transmitted wave. The electric field, at the output of twist, is vertical to slab of beef meat. A bidirectional coupler (H) is used to measure the forward and reflected waves. These data are used to determine lost power, including all components in addition to proposed material (D) [2].

Also, [3] presents a microwave freeze-drying setup in a laboratory scale.

5. Microwave energy and dielectric properties of different materials

According to what had been mentioned previously, the needed enthalpy for sublimation is provided by electromagnetic radiation at microwave frequency. Microwave heating is the result of the interaction between microwave fields and dielectric properties of material. In this heating, the required power for sublimation is carried by electromagnetic fields to the frozen region, independent of thermal characteristic of dried region, and then it is dissipated in frozen region and converted to heat. This means that any frozen point is a source of heat [3]. Compared to conventional freeze-drying process, penetrating electromagnetic fields and transferring the needed energy, independent of thermal characteristics of dried region, are the main factors in accelerating dehydrating process in microwave freeze-drying process [3]. Volumetric heating of frozen material accelerates drying process as fast as 75% [7].

For dielectric heating, some specific frequencies are released by the Federal Communications Commission (FCC) in microwave region: 913, 2450, and 5800 MHz [10, 11]. The 2450MHz is more common and used in home microwave oven [10].

Microwave radiation has unique advantages that make it suitable for food industry. The following are two of them:

- (a) Microwave radiation is nondestructive and non-ionization radiation; hence, it is safe and does not contaminate and deteriorate the material.
- (b) The electromagnetic waves are passed from dielectric materials in microwave frequencies, while these media are not transparent to light and infrared radiation (opaque). It is a useful tool for probing the dielectric material thoroughly [12]. This property is used in freeze-drying method to transfer energy inside the dielectric.

Also, microwave radiation is widely used in agriculture: remote sensing, short-range radars, and Doppler radar. Short-range radars are helpful for recognizing and tracking the seasonal

migration of birds and insects. Also, Doppler radar is used to monitor the mass flow rate of crop [12], detecting and controlling the insects in stored grains and heating seeds with impermeable coat by microwave radiation to improve their germination and determination of moisture content of agricultural products [12].

Microwave energy is used to defrosting meat. It reduces the required time from hours to a few minutes. Also, it is used in sterilizing some heat-sensitive foods and cacao bean roasting [12].

In microscopic scale, when a dielectric is subjected to the electric field, their molecules are arranged to reduce the overall electric field in the bulk of dielectric (**Figure 8**). This arrangement depends on constitutive molecules and their polarizations. In other words, the molecules reacted to the applied field.

The molecules start to oscillate by applying the electric field with sinusoidal variation. Friction between molecules in the oscillation produces heat (as a thermal source) and increases the temperature of dielectric [7, 10]. Since the needed energy for oscillating molecules is provided by the electric field, the generated heat is as a result of energy conversion (from electromagnetic to thermal). Another phenomenon, which is effective in the loss factor of a dielectric, is ionic conduction. It relates to movement of dissolved ions, and the generation of heat when these ions collide with other molecules and atoms [10]. In a macroscopic view, this phenomenon is characterized by imaginary part of permittivity. Also, real part of permittivity is known as an ability of structure to be polarized.

Generally, the electromagnetic properties of each material are specified by relative electric permittivity (ϵ_r) and magnetic permeability (μ_r), both of them are complex quantities. The real and imaginary parts of ϵ_r , which are known as dielectric constant and loss factor, are related to stored and dissipated electrical energy in the material, respectively [12]:

$$\epsilon_r = \epsilon'_r - j\epsilon''_r, \tan(\delta) = \frac{\epsilon''_r}{\epsilon'_r}, p_d = \omega\epsilon_0\epsilon''_r |E|^2 \quad (4)$$

Figure 8. The arrangement of molecules when dielectric is subjected in external electric field.

where p_d is the volume density of dissipated power (W/m^3) and E is root-mean-square value of electric field. This is the rate of absorbed microwave energy which is converted to heat. In reality, these parameters must be replaced in the right-hand side of Eqs. (1) and (2) [10].

The dissipated power is proportional to ϵ'' and square of applied electric field. The more ϵ'' , the more the dissipated power. In freeze-drying process, the dissipation factor of frozen meat is much more than a dried meat, so the dissipated power in the frozen region is much more than a dried region. The dissipated power can be maximized if the material is located in where the electric field is maximum. The more ϵ' , the more the ability in polarization for a dielectric.

To obtain the electromagnetic fields formed in foodstuff, Maxwell's equations must be solved. They depend on the cavity, dielectric property, and geometry of material [10]. As known, Maxwell's equations are a set of partial differential equations that are coupled to each other:

$$\vec{\nabla} \times \vec{E} = -\frac{\partial \vec{B}}{\partial t}, \vec{\nabla} \times \vec{H} = \vec{J} + \frac{\partial \vec{D}}{\partial t}, \vec{\nabla} \cdot \vec{D} = \rho, \vec{\nabla} \cdot \vec{B} = 0, \vec{D} = \epsilon_0 \epsilon_r \vec{E}, \vec{B} = \mu_0 \mu_r \vec{H} \quad (5)$$

The last two equations determine the interaction of electromagnetic fields with matter. By considering the boundary conditions, solving these equations determines electromagnetic fields.

As mentioned previously, microwave energy is needed to heat the frozen material in microwave freeze-drying method. Accordingly, a microwave system must be designed to generate and deliver microwave energy to an applicator in the chamber. Therefore, each microwave system consists of three parts, source, applicator, and transmission media, for transferring power from the source to the applicator [13]. The applicator plays the role of a load in this system. Usually, waveguides and their components are used to provide transmission media.

Typically, vacuum tubes are used for generating high power at microwave frequencies. They include klystron, magnetron, traveling-wave tube (TWT), and so on. The magnetron is a more common tube which is used in the industry and home applications [13]. The following figure shows the position of magnetron among other microwave sources.

The output power of magnetron is usually ranged between 500 and 1500 W for home microwave oven [14]. For industry application, this power is up to 10 KW and more [7, 13, 15] (**Figure 9**).

Generally, the mechanism of a vacuum tube is due to the interaction between electromagnetic field and electron beam inside a vacuum envelope (**Figure 10**) [14]. For magnetron, the envelope consists of multi-cavity resonators along the peripheral of a cylinder. An electron gun, coincided with the cylindrical axis of the tube, produces an electron beam in the magnetron. In practice, energy is transferred to the electromagnetic field through electron beam, after electron bunching occurs. The way for output power can be provided through a probe, loop, or window [14].

The efficiency of microwave oven is less than 50%, which is more than conventional heating [14]. For modern magnetrons, this parameter is 70% [13, 16] (**Figure 11**).

Figure 9. The position of magnetron among other microwave sources (reproduced from [14]).

Figure 10. Top view of a magnetron (reproduced from [13]).

Figure 11. A microwave oven (reproduced from [14]).

The waveguide is a hollow conductor with uniform cross section, usually rectangular and circular, that is a mediator between source and load for transferring energy. **Figure 12** shows the rectangular and circular waveguides.

The energy transfer in waveguides is carried out in a form of propagating modes. To determine different modes in a waveguide, Maxwell's equations must be solved with respect to necessary boundary conditions (zero tangential electric fields in the sidewalls of waveguide). In reality, each mode determines the distribution of electric and magnetic fields in the transverse plane. Each mode is characterized by its cutoff frequency (f_c). For frequencies higher than f_c , the mode propagates inside the waveguide, and the mode is evanescent for frequencies lower than f_c [14]. Hence, the waveguides are known as high-pass structures. The mode with the lowest f_c is known as dominant mode. From the lowest to highest frequencies, this mode is the first mode that is propagated in the waveguide. With increasing frequency, other modes are excited in the waveguide. This state is called overmode [14]. Usually, each waveguide is used in a dominant mode. The dominant mode is TE_{10} for rectangular waveguide and is TE_{11} for circular waveguide.

Among other guiding-wave structures, waveguide has low loss property in high frequency, which enables it to carry very high power.

According to the above discussions, a wave can be propagated along the waveguide. When the open end of waveguide is shorted, the power is reflected back (to the source), and standing wave is formed in the waveguide. A cavity can be made by blocking both ends of a section of

Figure 12. Rectangular and circular waveguides.

waveguide. This structure is capable to store energy at specific frequencies. These frequencies are determined by characteristic equation. For dissipating the stored energy, it is enough to fill the cavity with lossy material. The cavity is excited by a probe, a loop, or an aperture in its sidewall. The excited fields are achieved by solving Maxwell's equations with respect to boundary conditions. The boundary conditions are zero tangential electric fields in all sidewalls of the cavity.

Dependent on the electrical length of different dimensions of the cavity, the cavities are divided into two categories: mono-mode and multimode cavities. The mono-mode cavity has small dimensions, in the range of wavelength, so that the single mode is excited inside it [13]. Therefore, this cavity is not suitable for big-volume material [13]. The excited mode is created at certain frequency because of resonant nature of mono-mode cavity. For maximizing absorbed power, the foodstuff must be located in the position(s) of the maximum electric field. Since maximum electric field occurs in one or a few positions, then the single-mode cavity is suitable for material with high loss. Increasing the dimensions of the cavity causes the excitation of different modes simultaneously. The excitation of different modes is dependent on cavity dimensions, the location of material and its electrical characterizations, and so on. The excitation of multimodes results on nonuniform field distribution in the cavity and non-uniform heating absorption in turn [13].

In freeze-drying method, frozen material is placed inside a cavity. The cavity is excited, and microwave energy is dissipated by material to provide needed energy for sublimation. In other words, the material absorbs microwave energy (a sink for microwave energy), and it is similar to a load for microwave system. Some stubs are used to match the load and prevent the reflecting waves coming back to the source.

Different methods are developed for measuring the permittivity of material. One of them uses resonant cavity to measure the permittivity. It is used in microwave frequencies and is based on perturbation theory. In this method, a small volume of material (relative to volume of the cavity) is placed in the point where the electric field is maximum. The existence of material in the cavity is equal to change in resonant frequency of the cavity [14]:

$$\frac{\omega - \omega_0}{\omega_0} \approx - \frac{\int_{V_0} (\Delta\epsilon |E_0|^2 + \Delta\mu |H_0|^2) dv}{\int_{V_0} (\epsilon |E_0|^2 + \mu |H_0|^2) dv} \quad (6)$$

Subscript 0 indicates the parameters of the cavity in the absence of material. Another method uses open-ended transmission line. This structure behaves as a resonator at microwave frequency. Different parameters affect the resonant frequency of this structure such as the length of line and fringing fields in the open end of line. The existence of different materials in the open end of line changes the fringing fields. In equivalent circuit, these fields are modeled by a capacitor (**Figure 13**). Its capacitance is affected by the medium at the open end of line. Changes in resonant frequency are due to permittivity of the proposed material, provided that other key factors do not change [12].

For low frequency, it can be possible to use a capacitor for measuring dielectric permittivity of material. In this method, the proposed material is used as a dielectric for the capacitor. It is sufficient that this capacitor is connected to a sinusoidal signal generator with a resistor in a series of combination. Now, the voltage dropped through the capacitor is measured in two

Figure 13. Open-ended transmission line and its equivalent circuit (reproduced from [12]).

cases: a capacitor with and without the proposed material as the insulator of capacitor. The change in dropped voltage depends on the change in capacitance. Accordingly, the change in capacitance of capacitor is related to the permittivity of material.

While for most of agricultural products, μ_r is assumed to be the unity, since most of the foodstuffs are nonmagnetic products [10], the permittivity for each material depends on its moisture content, operating frequency, temperature, etc. [12]. Different models have been proposed to follow the frequency variation of a dielectric such as Drude, Debye, Lorentz and Cole-Cole models [10].

For food products, the chemical compositions such as salt play a decisive role in dielectric properties of products [10]. Also, dielectric properties can be affected from physical properties, such as bulk density, particle size, and homogeneity [10]. For hygroscopic material, the water content is the most important factor in the dielectric constant [10].

The dielectric constant for water is 78 at room temperature. For the moist food, this parameter ranges between 50 and 70, and their loss factor is less than 25 [10]. For organic constituents in food material, the dielectric constant and loss factor are less than 3 and less than 0.1, respectively [10, 17].

Researches and studies have shown that water is the most important part which absorbs microwave energy in foodstuff [10, 17–19]. The dielectric property of water is listed in **Table 1** for different frequencies.

Table 2 lists the dielectric properties of some fruit and vegetable in two frequencies.

Frequency (MHz)		0.6	1.7	3	4.6	7.7	9.1	12.5	17.4	26.8	36.4
ϵ'	$T = 20^\circ\text{C}$	80.3	79.2	77.4	74	67.4	63	53.6	42	26.5	17.6
	$T = 50^\circ\text{C}$	69.9	69.7	68.4	68.5	67.2	65.5	61.5	56.3	44.2	34.3
ϵ''	$T = 20^\circ\text{C}$	2.75	7.9	13	18.8	28.2	31.5	35.5	37.1	33.9	28.8
	$T = 50^\circ\text{C}$	12.5	3.6	5.8	9.4	14.5	16.5	21.4	27.2	32	32.6

Table 1. The dielectric property of water [10, 20].

Fruit/vegetable		Apple	Banana	Carrot	Grape	Mango	Orange	Potato	Strawberry
Moisture content (%) (wet basis)		88	78	87	82	86	87	79	92
Tissue density (g.cm ⁻³)		0.76	0.94	0.99	1.1	0.96	0.92	1.03	0.76
ϵ'	915 MHz	57	64	59	69	64	73	62	73
	2450 MHz	54	60	56	65	61	69	57	71
ϵ''	915 MHz	8	19	18	15	13	14	22	14
	2450 MHz	10	18	15	17	14	16	17	14

Table 2. The dielectric properties of some fruits and vegetables in two frequencies [10, 15].

Ref. [10] is a valuable and comprehensive paper that reviews many references and collects (and integrates) diverse information about dielectric property of different agriculture and food materials (flour, dough, milk, cheese, nuts, fish, seafood, meat). More discussions are available on how to change the dielectric property for different materials [10].

6. Conclusion

The freeze-drying method is described in this chapter. Different relations, related to heat and mass transfer in the system, are mentioned. Time variation of temperature in different layers of system is shown. Then, the interaction between electromagnetic field and molecules of material is expressed. Dielectric properties of different foodstuffs are presented from different references. Different parts of a microwave system are fairly explained, too.

The final stage of freeze-drying method is time-consuming when the frozen region is disappearing. This method is costly and affordable for high-value food like coffee.

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