CHAPTER 3

DIELECTRIC PROPERTIES OF CEMENT AND CONCRETE

The use of microwave energy to improve the properties of cement-based materials is a relatively new area of research. However, this is a growing area of interest because microwave heating has many advantages, including high speed for heat generation, highenergy penetration, instantaneous and precise electronic control, and clean process. Due to various conventional curing methods have many limitations. For example, they take a long time to reach the strength required for water curing [71]. In addition, due to the inherent thermal insulation of concrete, they have non-uniform hydration products [69]; this causes different temperatures to occur in processed concrete under high-stream and temperature-curing conditions. Therefore, numerous research studies have clearly indicated that curing by microwave can further increase the early-age strength of cementitious materials [26,115]. However, some aspects have not been taken into account. Thus, this chapter presents an investigation the dielectric permittivity of cement-based materials during the initial period of hydration at a frequency of 2.45 GHz.

3.1 Fundamental of the dielectric properties of microwave and cement-based materials

The term microwave heating is equally applicable to microwave systems in both cases; for both, the heating is due to the fact that dielectric material, material with small but finite electrical conductivity, absorbs energy when it is placed in a high-frequency electric field. Consequently, electrical dipole polarization and conduction will be generated within dielectric materials, which are composed of polar molecules with positive and negative poles. These orderly dispersed polar molecules vibrate instantaneously and violently in correspondence to the alternative high-frequency electric field of microwave. It is necessary to overcome the resistance of molecular attraction and motion, because when friction generates heat, the temperature of the material is evaluated simultaneously.

Based on Lambert's law, the microwave energy absorbed can be defined as the rootmean-square value of the electric field intensity \vec{E} is normally used to evaluate the microwave energy absorbed in Eq. (3.1) [27].

$$Q = \sigma \left| \vec{E} \right|^2 = 2\pi f \varepsilon_0 \varepsilon_r \left(\tan \delta \right) E^2$$
(3.1)

where Q is the microwave energy, σ is the effective conductivity, f is the frequency, ε_0 is the permittivity of free space (8.8514 x 10⁻¹² Farad/meter), ε'_r is the relative dielectric constant, tan δ is the loss tangent coefficient, and \vec{E} is the electric field intensity.

Since concrete is a dielectric material, its intrinsic properties affect the way it interacts with the electric and magnetic fields of the microwaves. Dielectric property can be characterized by two independent electromagnetic properties: the complex (electric) permittivity ε^* and the complex (magnetic) permeability μ^* . However, most common concrete materials are non-magnetic and yield permeability μ^* that is very close to the permeability of free space ($\mu_o = 4\pi x 10^{-7} Henry/meter$) [16]. Thus, this study focuses on the complex ε^* , which comprises real and imaginary parts as the relationship expressed in Eq. (3.2):

$$\varepsilon_r^* = \varepsilon_r' - \bar{j}\varepsilon_r'' \tag{3.2}$$

where ε'_r and ε''_r are the real part and imaginary parts respectively, of the complex permittivity, and $\overline{j} = \sqrt{-1}$.

The real part of the relative complex permittivity, referred to as the dielectric constant, ε'_r , measures how much energy transferred from an external electric field is stored in a material. The imaginary one, ε''_r , measures how lossy a material is to an external electric field and is referred to as the relative loss factor. Moreover, an essential ratio that shows the energy lost (relative loss factor) to the energy stored (relative dielectric constant) in a material is given as loss tangent tan δ , as shown in Eq. (3.3):

$$\tan \delta = \varepsilon_r'' / \varepsilon_r' \tag{3.3}$$

As discussed earlier the dielectric property plays an important role to describe the behavior of concrete materials when subjected to microwave energy. Research works have been investigated continuously on basic dielectric properties [16,30,32]. However, some aspects have not been taken into account. Thus, this paper investigates the dielectric permittivity of cement-based materials during the initial period of hydration at a frequency of 2.45 GHz. The investigation uses a network analyzer with a transmission line technique based on the influences of water-to-cementitious ratios, cement types, pozzolan materials, and aggregates.

3.2 Materials and methods

3.2.1 Materials used

Types I and III hydraulic Portland cements were used throughout this test. Their chemical composition and physical properties are shown in Table 3.1. The ASTM C 618 classifies the PFA as low calcium (Type F). Silica fume (hereinafter referred to as SF) was also used as a high-pozzolanic material in accordance with the ASTM C 1240.

Deionized water with a pH 7.5 and river sand with a fineness modulus of 2.58 and gradation conforming to the ASTM C 33 were mixed in specific proportions. The chemical admixture used superplasticizer that conforms to the ASTM C 494; that is, the superplasticizer had a recommended dosage rate of 1000 ml per 100 of a kilogram of cementitious materials. Mineral admixtures included pulverized fuel ash (here-in-after referred to as FA) from an electricity power plant that used lignite coal as a raw material to produce a combustion-yielding steam for driving the generator.

Materials	SiO ₂	:Al ₂ O ₃	. CaO	Fe ₂ O ₃ -	, MgO -	K ₂ O	Na ₂ O	-SO _{3 1}	-L.O.I
OPC	20.30	5.67	60.43	6.23	3.14	0.90	0.36	2.80	2.80
FA,	47.80	22.70	2.18	21.90	0.92	2.15	0.32	0.37	1.44
MK	55.14	43.95	0.08	0.75	0.40	2.10	0.03	0.05	12.50
SF	96.0	0.10	0.12	0.10	0.13	0.45	0.10	-	2.57

 Table 3.1 Chemical composition of the materials used (% by mass).

Materials	Specific Gravity	Specific Surface Area (BET method) (m ² /g)
OPC	3.12	0.85
FA	2.45	0.70
МК	2.25	0.60
SF	2.90	18.02

 Table 3.2 Specific gravity and surface area of the materials used.

The sizes of the Portland cement and FA particles as shown in Fig. 3.1 were obtained using a micrograph sedigraph instrument. The method is based on X-Ray attenuation of a power, which is suspended in a sample cell and allowed to settle. Studies performed using the Malvern instrument Mastersizer 2000 particle size analyzer have achieved accuracy of $\pm 1\%$ as designated by the Dv50 using the Malvern Quality Audit Standard.



Diameter (µm)

(a) Portland cement Type I OPC and pulverized fuel ash (FA)





(b) Silica fume (SF)

Fig. 3.1 (Cont.) Particle size distributions of Type I Portland cement OPC and FA.

Deionized water with a pH value between 7.0 and 7.5 was used. Ottawa sand, which has a fineness modulus equal to 2.58, constitutes a fine aggregate accurately graded to pass a 850- μ (US Standard No. 20) sieve and to be retained on a 600- μ (US Standard No. 30) sieve—both of which are used to test cements. The primary reason for using Ottawa sand is its the quality: its grains do not show visible signs of clay, loam, iron compounds, or other foreign substances. Because the sand is typically regarded as a standard, it is often not subjected to detailed mineralogical analyses prior to experiments. However, for this study, we did examine a lab-grade sample in order to identify any impurities present. Crushed limestone rock with a fineness modulus and nominal maximum size equal to 5.74 and 6.0 cm respectively was used as coarse aggregate. Moreover, the test results of specific gravity and absorption of aggregates are also listed in Table 3.3.

Table 3.3	Specific	gravity and	l absorption	of aggregates
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Properties	crushed limestone rock	river sand
Bulk Specific Gravity (SSD basis)	2.72	2.34
Bulk Specific Gravity (Oven-Dried basis)	2.71	2.42
Apparent Specific Gravity (SSD basis)	2.70	2.40
Absorption (%)	0.60	0.81

Chemical admixtures: Well-known commercially as Sikament 520 High Range Water Reducing Admixture, the superplasticizer conforms to the ASTM C 494, as comply with ASTM C 494 Type G. Produced and distributed by the Sika Company, Ltd., this particular superplasticizer does not contain calcium chloride or any other intentionally added chlorides, and it will not initiate or promote the corrosion of steel reinforcement in concrete. For general concrete applications, Sika recommends a dosage rate of between 500 – 1200 ml/100 kg) cementitious materials. If maximum water reduction is required, a dosage up to (1400 ml/100 kg) of cementitious materials can be used. In this case, delayed setting times may occur.

3.2.2 Mixture proportions

The proportions of cementitious materials mixed in preparation for specimen testing are shown in Table 3.4.

 Table 3.4 Mixtures used (by weight in grams).

Group	Factors affecting	Symbol	W/S-	OPC	FA	SF	MK	Water.	Superplasticizer Type G	Sand	Rock
and the second			記録	(ġ)	(g)	.(g) (: (g)		(CC)	s (g)	, (g) (
	1 Water-to-solid	1CW/S_P0.25	0.25	500	0	0	0	125	0	0	0
1		1CW/S P0.38	0.38	500	0	0	0	190	0	0	0
¹ mass ratio, w/s	1CW/S_P0.45	0.45	500	0	0	0	225	· 0	0	0	
<u> </u>	· · · · · · · · · · · · · · · · · · ·		0.38	400	100	0	0	190	5	0	0
2	Pozzolan	2CAP 0.38MK20_Super_10%	0.38	400	0	0	100	190	5	0	0
		2CAP 0.38SF20 Super_10%	0.38	400	0	100	0	190	5	0	0
	Aggregate, Mortar	3CAM0.38_1:2.75	0.38	500	0	0	0	0	5	1375	0
3	Aggregate, Concrete	3CAC0.38_1:1:1	0.38	500	0	0	0	0	5	500	500

3.2.3 Testing procedures

In order to measure the dielectric properties of the paste, mortar, and concrete, it was necessary to use a vector network analyzer (VNA). To measure the dielectric properties of cementitious materials at a frequency of 2.45 GHz, a network analyzer with an open-ended coaxial probe, as shown in Fig. 3.2, was used. The analyzer consisted of a coaxial cavity; microwave reflectometer; 3.5-mm coaxial cable; 3.5-mm female calibration; and short-, open-, matched-load software. The coaxial cavity characterizes measurement in the range of 1.5 - 2.6 GHz with precision not more than 2% of the dielectric constant and 5% of the dielectric loss factor. The measured sample should be assumed; i.e., it should be assumed to have infinite size, non-magnetic material, isotropic and homogeneous properties. In addition, the coaxial cavity must be in contact with the sample under test (MUT). The Nicholson-Ross-Weir conversion process [21] was used to calculated dielectric properties. After the cementitious material had been mixed and placed in the mold, it was wrapped in Styrofoam that was 5.0 mm thick in order to protect it from heat loss. Both dielectric properties and semi-adiabatic temperature using a data logger with thermo-couple (Type K) was simultaneously recorded every 180 and 15 minutes, respectively. However, in order to eliminate the effect of the thermo-couple embedded in microwave radiation, three samples were separately tested for dielectric properties and three for temperature rise.



Fig. 3.2 A network analyzer (open ended probe technique) (R = reflected power, T = transmitted power).

A measurement using the open ended probe technique involves measuring the reflected (S_{11}) and the transmitted signal (S_{21}) . The relevant scattering parameters relate

closely to the complex permittivity and permeability of the material by equations. The conversion of s-parameters to complex dielectric parameters is computed by solving the equations using a Nicholson-Ross-Weir (NRW) technique.

In order to obtain the permittivity and permeability from s-parameters, the Nicholson-Ross-Weir (NRW) technique (Fig. 3.3) was used to provide a direct calculation of both the permittivity and permeability from the s-parameters. The NRW technique is divergent at integral multiples one-half wavelength in the sample. This is due to the fact that at a point corresponding to the one-half wavelength, the s-parameter (S₁₁) gets very small. For a small s-parameter (S₁₁) value the uncertainty in the measurement of the phase of S₁₁ on the VNA is very large. Therefore, the uncertainty causes a divergence at these frequencies. Such divergences can be avoided by reducing the sample length, but it is difficult to determine the appropriate sample length when the sample's ε and μ are unknown. Measurement of the reflection coefficient and transmission coefficient requires a pair (S₁₁, S₂₁) of s-parameters of the material to be measured. However, due to phase ambiguity, the technique diverges for low-loss materials at frequencies corresponding to integer multiples of one-half wavelength in the sample. Hence, it is restricted to optimum sample thickness of $\lambda_g/4$ and used preferably for short samples.



Fig. 3.3 Nicholson-Ross-Weir for calculating dielectric permittivity.

3.3 Results and discussion

All measured waveforms were obtained in the time domain and a frequency range from 1.7-2.6 GHz (S-band). They were then converted into the dielectric permittivity (dielectric constant, ε' and loss factor, ε'') were calculated. The effect of water-to-cement ratio (w/s), pozzolan materials, aggregate type (sand with and without crushed limestone rock on its properties was studied and also discussed in this section.

3.3.1 Scattering parameters

Theoretically, scattering parameters or S-parameters describes the electrical behavior of linear electrical networks when undergoing various steady state stimuli by small signals. They are mostly used for networks operating at radio frequency (RF) and microwave frequencies where signal power and energy considerations are more easily quantified than currents and voltages. Conventionally, s-parameters are defined for a given frequency and system impedance, and vary as a function of frequency as shown typically in Table 3.5, for this study it is assigned in S-band (2.45 GHz). In order to convert from obtained s-parameter from dielectric parameters using a network analyzer, Nicolson-Ross-Weir technique was used due to fast, non-iterative and for applicable to waveguides and coaxial line.

Presence of water is recognized as the primary and dominant factor surrounding the increase of ε' in concrete. Other influencing factors are: electromagnetic (EM) frequency, water-to-cement ratio, porosity, ions in pore solution which make the material behave as conducting dielectrics, and clay minerals of wide range of porosities and specific surfaces. Factors such as cement type, salt impregnation, presence of pulverized fuel ash and ground granulated blast furnace slag, and temperature during tests are reported to be of minor or negligible significance. Literature was scarcely reported on the effects of different types of dense aggregate on ε' . However it is believed that the effects should be insignificant because of the relatively small variations of aggregate density.

Frequency	Wavelength	IEEE Radar Band designation
1 - 2 GHz	30 - 15 cm	L Band
2 - 4 GHz	15 - 7.5 cm	S Band
4 - 8 GHz	7.5 - 3.75 cm	C Band
8 - 12 GHz	3.75 - 2.50 cm	X Band
12 - 18 GHz	2.5 - 1.67 cm	Ku Band
18 - 27 GHz	1.67 - 1.11 cm	K Band
27 - 40 GHz	11.1 - 7.5 mm	Ka Band
40 - 75 GHz		V Band
75 - 110 GHz		W Band
110 - 300 GHz		mm Band
300 - 3000 GHz		u mm Band

Table 3.5 IEEE Radar Band Designations.

As shown in Figs. 3.4 - 3.6, the values of S_{11} and S_{21} values from which were measured in the range of 1.7 - 2.6 GHz of cement pastes at a delay time of 30 minutes with different water-to-cement ratios (w/s), with different pozzolan materials and mortar and concrete are represented, respectively. In general, S_{11} and S_{21} represent reflection coefficient at port 1 (inlet signal) when port 2 (outlet signal) in terminated with a matched load and attenuation of wave travelling for port 1 to port 2, respectively.

For S_{11} and S_{21} values with different water-to-cement ratios (w/s), the tendency of S_{11} and S_{21} corresponds to the incident wave signal or sinusoidal wave behavior. It can be seen that cement pastes at w/s ratios of 0.38 and 0.45 have similar trend, while the 0.25 cement paste has different value, especially at frequency 1.7 - 1.9 GHz. This may due to the free water content of the paste 0.25 is lower than those of others pastes.



Fig. 3.4 S_{11} and S_{21} values of cement pastes with different water-to-cement ratios.



(d) S₂₁ imagination values

Fig. 3.4 (Cont.) S_{11} and S_{21} values of cement pastes with different water-to-cement ratios.



Fig. 3.5 $S_{11} \, \text{and} \, S_{21}$ values of cement pastes with different pozzolan materials.



Fig. 3.5 (Cont.) S_{11} and S_{21} values of cement pastes with different pozzolan materials.



Fig. 3.6 S_{11} and S_{21} values of mortar and concrete.



Fig. 3.6 (Cont.) S₁₁ and S₂₁ values of mortar and concrete.

3.3.2 Dielectric permittivity of the constituents of cementitious materials

Table 4.6 shows dielectric permittivity as a function of the temperature of all cementbased constituents. In general, dielectric permittivity consists of dielectric constant (ε') and loss factor (ε'') (Portland cement Types I and III, silica fume, pulverized fuel ash, superplasticizer, river sand, and crushed limestone rock) as a function of temperature change. It can be separated into three groups of materials including the following:

(a) The powder material group comprises Portland cement Types I and III, silica fume, and pulverized fuel ash. This group shows a narrow range of dielectric constant and loss tangent throughout the temperature range of 20 - 50 °C. However the loss factor of the

materials has a wide variation that is greater that its dielectric constant; this is due to ε' having a high variation and it is related to the conductivity of a material's polarization and relaxation behaviors.

(b) The water-based materials group consists of water and superplasticizer (polycarboxylic water-based). It has a reasonably wide range of both properties. This is because water is a dielectric material with ε' and $\tan \delta$ much higher than that of the other components (Metaxas, 1991). This effect shows clearly in chemical admixtures that consist mainly of water and polymer. In fact, the manufacturer specified that the solid content of superplasticizer is 40% by weight. Thus, the dielectric properties of admixtures are strongly influenced by water content.

(c) The aggregate materials group, for example, the fine aggregate (river sand) and the coarse aggregate (crushed limestone rock), has dielectric properties that are narrower than those of the powder and water-based material groups. For the dielectric constant under the same compaction condition, fine aggregate materials show a lower value than powder materials because the former is looser in its consistency.

Constituents	Dielectric const	ant $(\varepsilon'); \varepsilon'(t) = a$	$t^3 + bt^2 + ct + d$	
	$a \pm s:d$	$b \pm s.d$	$c \pm s.d$	$d\pm sd$
Portland cement Type I	-3.214E-07 ±	3.414E-05 ±	-2.220E-03 ±	3.215E+00 ±
	0.251E-06	0.915E-04	0.114E-03	0.731E-01
Portland cement Type III	-1.033E-07 ±	3.319E-05 ±	-1.311E-03 ±	6.841E+00 ±
	0.409E-06	0.881E-04	0.245E-03	1.377E-01
Pulverized fuel ash	3.614E-07 ±	-5.231E-05 ±	4.301E-03 ±	4.741E+00 ±
	2.702E-06	4.997E-04	1.224E-02	0.220E-01
Silica fume	-3.482E-07 ±	5.614E-05 ±	-3.140E-03 ±	2.785E+00 ±
	6.237E-08	2.005E-05	0.775E-03	0.678E-01
Superplasticizer	-2.415E-05 ±	3.995E-03 ±	-3.881E-01 ±	2.423E+01 ±
(polycarboxylic water-	7.995E-05	8.143E-03	0.235E-01	0.874E-01
based)				
Fine aggregate	-0.952E-08 ±	-2.155E-06 ±	2.808E-04 ±	2.119E+00 ±
(River sand)	5.020E-08	0.088E-05	0.214E-04	0.445E-01
Coarse aggregate	-7.223E-08 ±	2.448E-05 ±	-1.750E-03 ±	1.346E+00 ±
(Crushed limestone rock	1.414E-07	0.892E-05	0.223E-03	0.989E-01

Table 3.6 Dielectric constant (ε') and loss factor (ε'') of constituents

Constituents	Loss factor (ε'') ; $\varepsilon''(t) = et^3 + ft^2 + gt + h^3$					
	and the second of the second	$f \pm s.d.$		$h\pm s.d.$		
Portland cement Type I	2.874E-08 ±	-7.098E-06 ±	6.094E-04 ±	1.064E-01 ±		
	0.902E-08	0.098E-05	0.004E-04	1.134E-02		
Portland cement Type III	7.245E-08 ±	-3.948E-06 ±	6.904E-04 ±	3.033E-01 ±		
	0.885E-08	0.083E-06	2.312E-04	0.093E-02		
Pulverized fuel ash	-1.775E-07	2.098E-05	-1.349E-03	2.424E-01		
	0.975E-07	0.048E-05	0.904E-03	7.090E-02		
Silica fume	-3.028E-07 ±	6.438E-05 ±	-3.948E-03 ±	2.243E-01 ±		
	0.038E-07	0.048E-05	0.140E-03	0.3987E-02		
Superplasticizer	-2.208E-05 ±	3.039E-03 ±	-1.902E-01 ±	9.594E+00 ±		
(polycarboxylic water-	0.849E-05	0.088E-03	0.098E-02	1.759E+00		
based)						
Fine aggregate	7.008E-08 ±	-2.448E-05 ±	1.442E-03 ±	2.244E-01 ±		
(river sand)	0.084E-08	2.949E-06	0.233E-04	0.004E-2		
Coarse aggregate	8.044E-09 ±	-2.330E-06 ±	2.088E-04 ±	5.948E-02 ±		
(crushed limestone rock)	0.048E-09	8.904E-07	4.234E-05	0.740E-03		

Table 3.6 (Cont.) Dielectric constant (ε') and loss factor (ε'') of constituents

Remark: s.d. = Standard Deviation

3.3.3 Dielectric permittivity of pastes, mortars, and concretes

Fig. 3.7 shows the evolution of dielectric properties and the simultaneous temperature rise of cement pastes. It can be observed that the dielectric properties at the initial stage are relatively higher in comparison with the later stage; they also increase with the increasing water content (higher w/s) of the cement pastes. This is due to the fact that immediately after contact has been made between water and cement, they start to react and then Ca^{2+} , OH^{-,} and SO_4^{2-} ions dissolve into the system. In addition, during the dormant period, the dielectric properties change very little because the chemical composition of the aqueous phase remains nearly constant.

Similarly, relative dielectric properties also appear to be affected by the temperature rise. The lower temperature (higher w/s) leads to enhance these properties. This is due to a reduction in water-to-cement-ratio that accelerates hydration and results in higher temperature. Especially, in the accelerated period of the pastes at which it has the highest rise in temperature corresponding to the decreasing relative dielectric properties.



Fig. 3.7 Dielectric permittivity of pastes with different water-to-cement ratios.

Effects of silica fume (SF) and pulverized fuel ash (PFA) on the dielectric permittivity, temperature rise, and setting time of the pastes are shown in Fig. 3.8. The dielectric constant of the paste containing PFA through the first 24-hour hydration time is higher than that of the plain paste, whereas the paste containing SF is lower. Both reactions involving SF and PFA occur as secondary reactions. This means, however, that the PFA can produce excessive water in the paste while increasing Si^{3+} and Ca^{2+} ions in the system. This results in an increasing dielectric constant of the paste [19]. Unlike the SF paste, the compositions with PFA can dissolve it at a high rate; its fineness induces the bounding of its water molecules and ionrichness at the surface. As a result, the dielectric constant is lower than that of the plain cement paste. For loss factor evolution, the conventional paste and the SF paste show little difference in their comparative loss factors; however between, the difference in the loss factors of the conventional and SF paste when compared with the PFA paste is large. This indicates that the remaining water content, both during introduction and acceleratory periods in the paste, has a strong effect on dielectric loss. On the other hand, the PFA particles can retain free water in the paste system, which may cause the loss factor of the PFA paste to become very high.



Fig. 3.8 Dielectric permittivity of pastes with different pozzolan materials.

Fig. 3.9 shows that the relative permittivity curves for the mortar and cementitious material are also similar to the curve of the conventional pastes but lower than that of the pastes. The decrease of cement content and the absorption of water molecules by aggregate surfaces induce a lower concentration of ions in the system (Rhim and Buyukozturk, 1998). However, eventually, these parameters approach to constant. A comparison in the setting time range shows that the paste's permittivity decrease rate slope is somewhat higher than those of mortars and cementitious materials. This is due to the ion constraints of the hydrated products; specifically that the hydrated products have high ion concentration in comparison with the aggregate-mixed products.



Fig. 3.9 Dielectric permittivity of paste, mortar and concrete.

3.4 Conclusions of this section

3.4.1 The dielectric permittivity of cement-based materials can be classified into three groups; (a) the powder material group comprises Portland cement Types I and III, silica fume, and pulverized fuel ash. This group shows a narrow range of dielectric constant and loss tangent throughout the temperature range of 20 - 50 °C. (b) The water-based materials group consists of water and superplasticizer (polycarboxylic water-based). It has a reasonably wide range of both properties. (c) The aggregate materials group, the fine aggregate (river sand) and the coarse aggregate, has dielectric properties that are narrower than those of the powder and water-based material groups.

3.4.2 Dielectric permittivity of cement-based material is affected by initial water-tocement ratio, cement types, pozzolan and aggregate types. However, although the volumetric fraction of water and superplasticizer in a given mixture is small, it strongly affects the dielectric permittivity of the cement due to its high dielectric permittivity. The change in the dielectric permittivity is relatively high and remains constant during the dormant period; after that it decreases rapidly when the hydration reaction resumes, and it continues to decrease during the acceleratory period.