

Influence of CO₂ Concentration During Accelerated Carbonation Tests with Alternative Binders

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Abstract

One approach to improve the carbon footprint of concrete is the use of alternative binders, which can be produced with less CO₂ (Carbon dioxide) emissions and lower energy consumption. These alternative binders either have a reduced clinker to cement ratio or contain no Portland cement clinker at all. The protection of the embedded steel is an important characteristic, which determines the applicability of such binders in reinforced concrete structures. The progress of carbonation (Carbonation is a process that takes place at the nanometric scale) and the associated reduction of the pH value have a major influence in this regard, especially as these binders are typically less resistant against CO₂ exposure. Previous research has shown that accelerated carbonation tests at elevated CO₂ concentrations can lead to altered carbonation behavior, especially in systems low in calcium hydroxide. Accordingly, special consideration must be given to the CO₂ concentration in such accelerated tests and to the interpretation of the resulting carbonation behavior. In this study, the carbonation resistance of various alternative cements and binders, such as composite cements with calcined clays or modified steel slag, alkali-activated materials, CSA cement and a C-S-H binder were investigated on mortar samples stored in three different CO₂ concentration - atmospheric storage (~0.04 vol.%), 0.3 vol.% and 1.0 vol.% CO₂. The results were analyzed with regard to the carbonation progress and the specific carbonation behavior of the binders. It was found, that even within this comparably low range, the binders react very differently to an increased CO₂ concentration. Consequently, binder specific CO₂ concentrations might be necessary, if the results of accelerated tests are intended to reflect the carbonation behavior in atmospheric conditions.

Keywords

Alternative binders, Carbonation, Portland cement, Alkali-activated materials

Introduction

As part of the efforts to reduce the carbon footprint of concrete, the use of alternative binders is increasingly attracting attention. To be truly sustainable, these binders must not cause any reduction in the durability of concrete structures that is relevant to service life. In this context, the carbonation behavior, and the associated loss of corrosion protection of the steel reinforcement is a major challenge.

In general, carbonation of concrete is a comparably slow process. In practical conditions the carbonation depth after several years is typically in the range of a few Millimeters. Thus, the carbonation resistance of concretes is often investigated in accelerated test procedures, to gain results in relative short periods of

time. The accelerating effect is achieved by applying elevated CO₂ concentrations. As is known from other accelerated test procedures, e.g., from sulfate resistance tests [1], high concentrations of the attacking medium might lead to an alteration of the actual (damage) mechanism, resulting in misleading interpretations on the resistance of the tested materials.

For accelerated carbonation tests, the European standard DIN EN 12390-12 specifies a test concentration of 3.0 vol.% CO₂, which is about 75 times the natural CO₂ concentration in the atmosphere. For Portland cement-based concretes this concentration can seemingly be applied to accelerate the carbonation process without altering the actual mechanisms drastically. CO₂ concentrations above that value can lead to the formation of carbonation products, which do not occur in materials undergoing natural carbonation [2, 3].

For clinker reduced cements and for binders without Portland cement the critical threshold value is seemingly located in a much lower range, probably ≤ 1.0 vol.% CO₂ [4-7]. Hydrated systems of such binders contain lower amounts of Portlandite or no Portlandite at all. In these materials the main hydration phases such as C-S-H, C-A-S-H or C-(N)-A-S-H will undergo carbonation more rapidly [2, 8], leading to a coarsening of the pore structure. Obviously, these materials react much more sensitive to elevated CO₂ concentrations.

Consequently, the carbonation resistance of various alternative cements and binders, such as composite cements with calcined clays or modified steel slag, alkali-activated materials (on the basis of Ground Granulated Blast Furnace Slag (GGBFS) or Metakaolin), CSA cement and a C-S-H binder was investigated in this study. The experiments were carried out on mortar samples stored in three different CO₂ concentrations - atmospheric storage (~0.04 vol.%), 0.3 vol.% and 1.0 vol.% CO₂. The results were analyzed with regard to the carbonation progress and the specific carbonation behavior of the binders.

Materials and Method

Materials and Composition

This study covers a wide range of alternative binders - from clinker-reduced cements containing various supplementary cementitious materials (SCMs) to Portland cement clinker-free binders, such as calcium sulfoaluminate (CSA) cement, calcium silicate hydrate (C-S-H) binders, and alkali-activated materials (AAMs). Additionally, two commercial cements were tested as reference materials. Table 1 gives an overview on the binders and the applied water/binder-ratios (w/b-ratios).

When comparing the performance of different cements, the w/b-ratios are typically kept constant for all investigated cements. In the present study it was not reasonable to follow this approach completely, as the concept of some binders differs strongly from Portland cement-based binders. The w/b-ratio of the alkali-activated binders is calculated from the added water plus the liquid portion of the activator. Since Celitement® forms exclusively C-S-H-phases, the w/b value must be assessed differently than for the other binders and is

Table 1: Overview of the binders and w/b ratios.

Group	Identifier	Description	w/b
Reference	Ref I	CEM I 42.5 R	0.50
	Ref II	CEM III/B 42.5 N	0.50
SCMs	CC I	30% Metacellite, 70% Ref I	0.50
	CC II	30% Metakaolin, 70% Ref I	0.50
	LD	30% steel slag, 70% Ref I	0.49
C \bar{S} A	C \bar{S} A	CSA with tartaric acid as retarder	0.50
C-S-H	C-S-H	Celitement®	0.40
AAMs	Geo MK	Metakaolin activated by potassium silicate	0.50
	AAS I	GGBFS I activated by sodium silicate	0.38

therefore lower. It was produced with the addition of a superplasticizer to ensure the necessary workability.

The reference cements used were CEM I 42.5 R (Ref I) and CEM III/B 42.5 N-LH/SR (na) (Ref II). The latter is based on the same clinker and contains between 65 and 80 wt.% GGBFS; the exact GGBFS content is unknown. Ref I consists of about 50% C₃S and about 20% C₂S, while C₃A accounts for about 5%. Ref II was also used to produce composite cements in a 70/30 ratio with calcined clays and with steel slag, respectively. The calcined clays used were a self-made metacellite and a commercially available metakaolin. The calcined clay used for CCI mainly contains quartz and X-ray amorphous phases, while the metakaolin (calcined clay used in CC II) consists almost exclusively of X-ray amorphous phases. The steel slag was a modified LD (Linz-Donawitz) slag. Half of the modified LD slag consists of C₂S. Other notable constituents (> 5%) are bredigite (Ca₇Mg[SiO₄]₄), wüstite ([Mg, Fe]O) and dicalcium ferrite (CaO Fe₂O₃ in short: C₂F). The C \bar{S} A was a commercially available product with tartaric acid as a retarder. It contains about 47% ye'elemite. Other constituents are anhydrite (24%), C₂S (16%), and C₃A (7%). The C-S-H binder is a pre-hydrated binder. By means of X-ray diffraction, an X-ray amorphous fraction of about 88% was determined. Another phase constituent is quartz. A metakaolin based geopolymer was prepared with potassium water glass as activator (silicate modulus 1.1). AAS I was activated with sodium silicate (silicate modulus 2.0). The metakaolin used for the geopolymer is 30% X-ray amorphous, the main component with about 70% is quartz. GGBFS I is, as expected, completely X-ray amorphous. Detailed material and hydration/polymerization analyses can be found in [7].

Investigation of carbonation depth due to natural and accelerated carbonation

Investigations on carbonation resistance were carried out on mortar prisms (4 x 4 x 16 cm³) produced analogously to DIN EN 196-1 (2016-11), but with w/b ratios as given above. The specimens with alkali-activated binders (AAS II and Geo MK) were stored at laboratory climate after demolding, as water storage at a very early hydration stage can lead to

dissolution phenomena at the edges of the sample. All other samples were stored underwater until 28 days of age. To determine the depth of carbonation by “natural” carbonation, the specimens were stored in standard laboratory climate at 20 °C and 65% relative humidity after the age of 28 days.

In preparation for accelerated carbonation, the mortar prisms were allowed to dry in laboratory climate (20 °C and 65% relative humidity) for a period of 14 days. Then, they were transferred to climate chambers with 20 °C and 65% relative humidity at 0.3 and 1.0 vol.% CO₂, respectively. To determine the progress of carbonation, the specimens were split at different time intervals (see table 2), and the fresh surfaces were sprayed with 1% phenolphthalein solution. The sprayed fracture surfaces were photographed and graphically analyzed using ImageJ image processing software.

The times referred to as test duration in the following sections are based on the beginning of the CO₂ exposure. Accordingly, the samples for natural carbonation were 28 days old at the beginning of exposure and those for accelerated carbonation had an age of 42 days. Regardless of the previous storage conditions, these times also apply to AAS I and Geo MK.

For the determination of the carbonation depth, it is assumed, as is usual for Portland cement, that the carbonation results in a change of the pH value, which is indicated by the color change of the indicator solution. Furthermore, the carbonation coefficient k_c was determined in accordance with DIN EN 12390-12 (2020-01) with the carbonation depths measured for the different exposure types. The carbonation depths were plotted against \sqrt{t} . The slopes of the linear regression of the measured values represent the carbonation coefficients.

Table 2: Carbonation testing - time intervals.

Time interval for testing the carbonation depth in d										
Atm CO ₂	0	7	14	22	28	56	84	112	140	175
0.3 vol.% CO ₂	0	28	56	84						
1.0 vol.% CO ₂	0	7	28	56	98	182	356			

Results and Discussion

First, the results of the carbonation depth measurements will be presented, and the effects of the exposure conditions will be discussed. The results of selected carbonation depths are shown in table 2.

The progress of the carbonation depth over the test duration is only presented exemplary for Ref I, Ref II, CSA, and AAS I, see figure 1. Please note that for Ref I the y-axis has a smaller scale than for the other three mortars.

As expected, all mortars, independent of the binder type, show clear differences between storage at atmospheric CO₂ and at 1.0 vol.% CO₂, as a strong acceleration of the carbonation occurs with the increased CO₂ concentration. The AAS I mortar was completely carbonated after about 100 d in 1.0 vol.% CO₂, the mortar based on the geopolymer Geo MK

even after 28 d (not displayed in figure 1, values in table 1). Contrary to the findings of Gluth et al. [9] the carbonation resistance of these alkali activated systems is very low and thus raises the question whether a CO₂ concentration of 1.0 vol.% is not too high for these binder types. However, the overall carbonation resistance of the geopolymer sample was found to be extremely low, regardless of the CO₂ concentration selected. Even in atmospheric CO₂ complete carbonation occurred after 110 days. The geopolymer was mostly excluded from the further analysis, as its carbonation resistance lies in a totally different range than for the other binders. All other binders showed a better carbonation resistance. In general, the performance was better for Portlandite-rich mortar (e.g., Ref I) than for Portlandite-poor mortars (e.g., CC II, CSA).

Following DIN EN 12390-12 (2020-01), the carbonation coefficients for the different storage types were determined, see table 3. For this purpose, the carbonation depth was plotted over \sqrt{t} .

Geo MK is again not included. Since all mortars show almost linear regressions, it can be assumed that the carbonation follows the CSA-characteristic. The coefficient of determination R² is ≥ 0.90 for the majority of the data, with only a few exceptions.

To enable an assessment of the magnitude of the coefficients, the results of this study are compared with literature values. Leemann et al. [12] determined the carbonation coefficients for mortars with CEM I, CEM III/B, and CSA stored at atmospheric CO₂ and at 1.0 vol.% CO₂. The comparison with their data shows good agreement for the carbonation coefficients of Ref I and Ref II, both for natural and accelerated carbonation. However, their carbonation coefficients are lower by a factor of 10 than the results presented here for CSA.

Gluth et al. [9] summarize, among other things, the carbonation coefficients of alkali-activated slags from various investigations. The carbonation coefficients determined here for AAS I are in good agreement with the data evaluated by Gluth et al. for CO₂ concentrations of 1.0 vol.%.

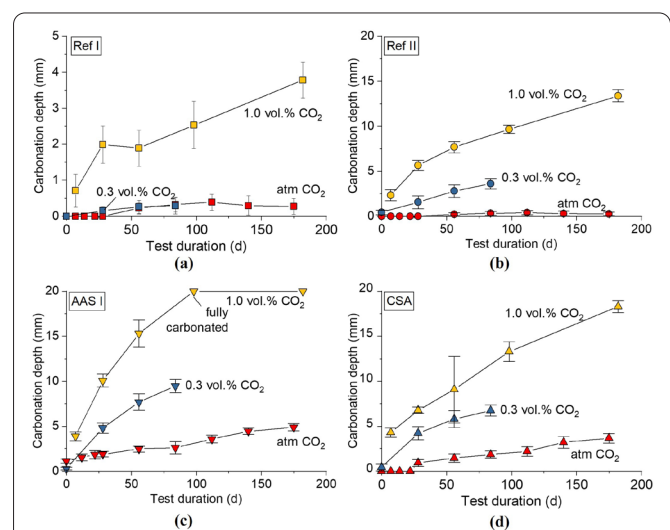


Figure 1: Carbonation depth versus test duration for natural carbonation (atm CO₂) and accelerated carbonation (0.3 vol.% CO₂ and 1.0 vol.% CO₂); (a) Ref I; (b) Ref II; (c) AAS I; and (d) CSA.

Table 3: Carbonation depth for natural (atm CO₂) and accelerated carbonation (0.3 vol.% CO₂ and 1.0 vol.% CO₂).

Carbonation depth in mm [*]										
	Dur. (d)	Ref I	LD	Ref II	CC I	CC II	C-S-H	C \bar{S} A	AAS I	Geo MK
Atm CO ₂	0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	1.13	5.02
	14	0.00	0.00	0.00	0.00	0.00	0.00	0.00	1.55	6.66
	28	0.00	0.42	0.53	0.38	0.00	0.27	0.94	1.91	8.21
	56	0.22	0.77	0.86	0.54	0.39	0.72	1.45	2.47	12.53
	84	0.32	0.63	0.91	0.81	0.58	0.93	1.87	2.61	15.30
	140	0.29	0.90	1.61	1.25	1.12	1.38	3.19	4.44	20.00
	175	0.27	0.63	1.59	1.23	1.09	1.65	3.66	4.89	20.00
Start value for acc. carbonation		0.00	0.19	0.43	0.19	0.18	0.70	0.43	0.31	1.17
0.3 vol.% CO ₂	28	0.16	0.42	1.55	0.91	0.60	1.09	4.22	4.80	20.00
	56	0.26	0.75	2.79	1.77	1.29	1.75	5.79	7.70	20.00
	84	0.29	0.71	3.60	1.86	1.39	1.97	6.75	9.47	20.00
1.0 vol.% CO ₂	7	0.71	1.49	2.33	2.04	1.41	2.46	4.30	3.88	11.82
	28	1.99	2.45	5.66	4.48	2.96	4.95	6.78	10.06	20.00
	56	1.89	3.04	7.67	5.84	3.79	8.43	9.07	15.29	20.00
	98	2.53	3.44	9.64	8.15	5.09	10.18	13.28	20.00	20.00
	182	3.78	3.80	13.38	11.39	7.10	13.07	18.29	20.00	20.00
	356	4.96	5.98	20.00	16.02	10.67	20.00	20.00	20.00	20.00

Note: ^{*}Due to the dimensions of the mortar prisms, the maximum, possible carbonation depth is 20.00 mm.

Trümer [11] and Kraft et al. [7] investigated the carbonation of calcined clays. They observed a higher carbonation coefficient for mixtures of metakolin than for mixtures of metakaolin, although the mixtures of metakolin had a higher Portlandite content [7, 11]. Both explain these differences by the fact that the hydration of the metakaolin-cement mixture results in a denser structure, which slows down the carbonation. Furthermore, Trümer [11] derives the coefficients for carbonation at atmospheric CO₂ from carbonation depths at 2 vol.% CO₂. These values are significantly higher than the coefficients determined here at lower CO₂ concentrations.

When distinguishing between carbonation by storage at atmospheric CO₂ and in 0.3 vol.% CO₂ the binders can be divided into three clusters (Table 4), in which the CO₂ concentration-dependent carbonation behavior is comparable, respectively. The first cluster contains Ref I and LD. For these binders there are only small differences between accelerated carbonation at 0.3 vol.% and atmospheric CO₂. The CO₂ content of 0.3 vol.% was thus too low to achieve a substantial accelerating effect. Furthermore, these two samples show the lowest carbonation depths for all exposures.

The second cluster includes Ref II, CC I, as well as CC II and C-S-H. It thus primarily contains the multicomponent

cements (Portland cement + SCM). These mortars show the expected acceleration of the carbonation progress at 0.3 vol.% CO₂, with carbonation depths ranging between the values obtained at atmospheric and 1.0 vol.% CO₂. Overall, these four binders show a significantly higher carbonation progress than Ref I and LD, however, the carbonation resistance is in line with previous experiences [7, 10, 11].

C \bar{S} A, AAS I and Geo MK represent the third cluster, making this cluster primarily composed of binders that do not contain any Portland cement clinker. These binders show a notable progress of carbonation already in atmospheric CO₂ concentration. In 0.3 vol.% CO₂ a strong increase of the carbonation occurs. In 1.0 vol.% CO₂ the mortars are completely or almost completely carbonated in the observed testing period. Likewise, these three binders reach the highest carbonation depths resp. are carbonated the fastest.

With reference to the amount of Portlandite formed during hydration/polymerization, it is noticeable that cluster 1 consists of the binders that form a relatively high amount of Portlandite (21 - 26 % after 28 days), while the binders from cluster 2 show a relatively low formation of Portlandite (6 - 13 % after 28 days). No formation of Portlandite at all was observed in the binders from cluster 3 (c.f. [7]). The C-S-

Table 4: Carbonation coefficients k_c for natural (atm CO₂) and accelerated carbonation (0.3 vol.% CO₂ and 1.0 vol.% CO₂) and coefficient of determination R².

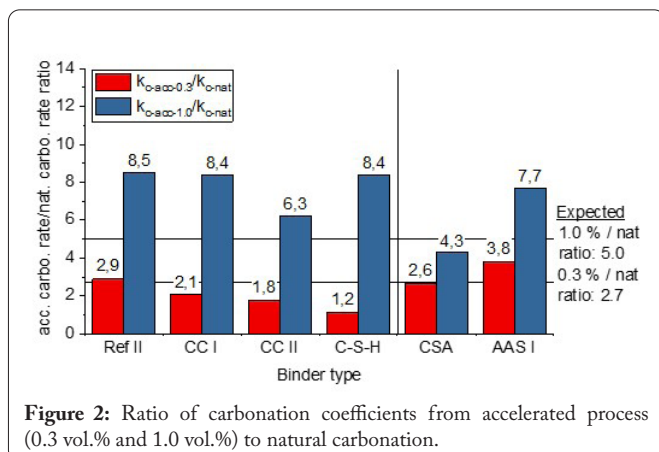
		Atm CO ₂		0.3 vol.% CO ₂		1.0 vol.% CO ₂	
		k_{c-nat} (mm/√d)	R ²	$k_{c-acc-0.3}$ (mm/√d)	R ²	$k_{c-acc-1.0}$ (mm/√d)	R ²
Cluster 1	Ref I	0.023	0.80	0.033	0.99	0.281	0.90
	LD	0.051	0.67	0.062	0.90	0.381	0.98
Cluster 2	Ref II	0.117	0.98	0.340	0.94	0.998	0.99
	CC I	0.092	0.98	0.192	0.94	0.774	1.00
	CC II	0.079	0.93	0.139	0.91	0.494	0.99
	C-S-H	0.121	0.97	0.140	0.91	1.015	0.96
Cluster 3	CSA	0.264	0.94	0.697	1.00	1.134	0.99
	AAS I	0.263	0.87	0.999	1.00	2.030	0.98

H-binder is an exception; although no significant amount of Portlandite is formed during hydration, it fits well into cluster 2 based on the carbonation depths. Another aspect that the binders from cluster 3 have in common compared to the binders from clusters 1 and 2 is that they form little to no C-S-H phases during hydration/polymerisation [7]. This gives an indication why mortars from C-S-H can be assigned to cluster 2 despite low portlandite content.

Of special interest is the difference of the carbonation coefficients for the individual CO₂ concentrations. This difference can be used to analyze, whether a critical alteration of the carbonation mechanism occurs for increased CO₂ concentrations. According to Sisomphon et al. [13] the relationship between carbonation coefficients of the accelerated and the natural process can be obtained from the following equation:

$$\frac{K_{c-acc}}{K_{c-nat}} = \sqrt{\frac{c_{CO_2 acc}}{c_{CO_2 nat}}} \tag{1}$$

For a 0.3% CO₂ concentration, the equation (1) becomes $k_{c-acc-0.3}/k_{c-nat} = 2.7$ and for a 1.0% concentration, $k_{c-acc-1.0}/k_{c-nat} = 5.0$. This means that for ratios above 2.7 or 5, the carbonation rate is overestimated by the accelerated method and vice versa. Figure 2 shows the results of $k_{c-acc-0.3}/k_{c-nat}$ and $k_{c-acc-1.0}/k_{c-nat}$ graphically.



Since the test durations of the natural carbonation were too short to be able to determine a significant carbonation progress for the binders of cluster 1, these samples will not be considered in the following. These data will be analysed in subsequent publications.

The ratio between $k_{c-acc-0.3}$ and k_{c-nat} is above 2.7 only for Ref II and AAS I, Ref II is the closest with 2.9. Consequently, the carbonation rate is overestimated for these mortars at 0.3 vol.% CO₂. All other mortars show a ratio of < 2.7, which implies an underestimation of the carbonation rate at 0.3 vol.% CO₂. Nonetheless, the deviations from the expected ratio are small, when compared with the deviations for 1.0 vol.% CO₂.

The ratio between $k_{c-acc-1.0}$ and k_{c-nat} is above 5 for all samples, except for CSA. This means that the accelerated method with 1.0 vol.% CO₂ overestimates the carbonation rate compared to natural carbonation. In other words, the carbonation is excessively accelerated in this concentration range. Only for CSA, the measured ratios are in good agreement with the expected ratios of the carbonation coefficients.

Finally, the ratios of the coefficients are compared with values from the literature, but it must be noted that very few data on CO₂ concentrations of 1.0 vol.% CO₂ and none on 0.3 vol.% CO₂ were found in the literature. In the summary by Gluth et al. [9], the data indicate that the resistance of blast furnace slag-rich cements is underestimated for CO₂ concentrations of 1.0 vol.%. This is in good agreement with the values determined here for Ref II. For mixtures of alkali-activated slags, the data used by Gluth et al. [9] deviate significantly in both directions from the expected value of 5.

Finally, it remains uncertain whether eq. (1) can be applied to alternative binders with low Portland cement contents over a broad range of CO₂ concentrations. It might be appropriate to define narrower, binder-specific ranges for the carbonation of alternative binders.

In general, the following aspects can be summarized for the three clusters described above:

- Cluster 1: Nearly no acceleration of carbonation was achieved with a CO₂ concentration of 0.3 vol.% CO₂, thus the application of this concentration for accelerated

processes is not reasonable.

- Cluster 2: Acceleration of carbonation is possible with both 0.3 and 1.0 vol.% CO₂. However, with 0.3 vol.% CO₂ the carbonation rate tends to be underestimated slightly compared to natural carbonation, whereas with 1.0 vol.-% it tends to be overestimated.
- Cluster 3: Since the mortars from this cluster already carbonate very strongly at atmospheric CO₂, accelerated carbonation is not necessary.

Conclusions

In this study, different alternative binders with and without Portland cement were investigated with regard to their carbonation behavior at atmospheric CO₂, as well as at 0.3 and 1.0 vol.% CO₂. The results and findings can be summarized as follows:

- In general, the carbonation of all binders used can be accelerated by storage at 1.0 vol.% CO₂. However, it remains questionable whether this acceleration leads to a misrepresentation of the mechanisms of natural carbonation.
- Storage at 0.3 vol.% CO₂ leads to an acceleration of carbonation only for mortars with a low Portlandite content (less than 20% after 28 days hydration). For binders with higher Portlandite contents no acceleration can be observed.
- Mortars can be divided into clusters characterized by different Portlandite and C-S-H phase contents and different reaction patterns to increased CO₂ concentrations.
- Assuming that the square root-of-time law holds, equation 1 shows that storage at 1.0 vol.% CO₂ overestimates the carbonation rate, while storage at 0.3 vol.% CO₂ tends to underestimate it. This applies for all binders except CSA cement.
- Geopolymer (Geo MK) carbonates so rapidly that no accelerated process is necessary.
- Overall, it should be considered that it may be useful to choose binder-specific CO₂ concentrations for accelerated processes.

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Conflict of Interest

None.

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