6A. Degradation of inorganic building materials

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6A. „degradation“ - content

- degradation (corrosion) agents
  - temperature
  - water
  - atmosphere
  - salts
  - biodegradation

- degradation of important building materials
  - aggregates, building stone, mortar, gypsum...
  - concrete
Degradation of building materials

- **gradual, spontaneous**, slow process in which material losses own characteristic properties due to environment.

- **corrosion environment** – external agents taking effect on the material (water – ground, rain, surface; temperature, chemicals in soil and air).

- Degradation of material: loss of strength and cohesion, dissolution, appearance..

- Corrosion of construction: **lifetime of construction**, reparation costs, loss of material, costs due to forced outage.

- **Prevention of corrosion**: selection of material (..concrete composition), protection of construction against aggressive environment.
Corrosion actions

- **physical** caused by physical forces, no chemical reactions
  
  *(frost damage of concrete)*

- **chemical** chemical reactions of material with environments components causing degradation of material
  
  *(sulphate degradation of concrete)*

- **biocorrosion** material is damaged by action of animals, plants and microorganisms and their metabolic products; *(dry rot)*
Chemical, physical and biological action often works **simultaneously** and **promote each other**

*Chemical reaction causes physical force (expansion of its products)*

*Bacteria produce aggresive chemicals*

*Mechanicaly damaged wall is easily chemicaly attacked*

*The degradation protection has to be complex, against all corrosion actions*
Corrosion action
Degradation due to temperature changes

- response of material to temperature changes:
  - very high temperature (fire): thermal decomposition of the material
  - „common temperature changes“ by cycling day-night, winter-summer and by sun shining (-20 to 40° C)

Thermal expansion: response of material to temperature changes $\rightarrow$ tension on binder+aggregates interface
$\rightarrow$ cracks

- loss of strength and cohesion, water attack to construction
Degradation due to temperature changes

- temperature gradient in construction

- temperature gradient
- different expansion of warm and cold parts of construction
Degradation due to water action

- water in building constructions

1. water vapor: component of atmosphere, everywhere higher temperature = higher possible absolute air humidity warm air may contain more vapor the the cold one relative humidity = degree of saturation by vapor at given temperature

2. equilibrium dampness: content of water in material depends on: air humidity, temperature, type of material is caused by vapor condensation in material a) bonded water – adsorbed on surface non-moveable, not dangerous, does not freeze
Degradation due to water action

- water in building constructions

2. equilibrium dampness: content of water in material
   b) free water: liquid in porous system of material
     origin: vapor condensation, ground water, rain
     movement in pores: up – by capillary action
       smaller pores = higher elevation
down – by gravity
     equilibrium of gravity and capillary
     elevation: cm to 2 meters above ground
Degradation due to water action

- **freezing-thawing action**: ice - 10 % higher volume than liquid water. Ice in pores expands and acts by **crystallization pressure** on the pore walls.
Degradation due to water action

- Water is solvent and transport medium for other corrosion agents

1. Anorganic and organic salts
2. Components of atmosphere (CO\textsubscript{2}, NO\textsubscript{x}, SO\textsubscript{x})
3. Soluble components of material: Ca(OH)\textsubscript{2}, CaSO\textsubscript{4} \cdot 2H\textsubscript{2}O

→ Chemical corrosion

- Water is necessary for life of bacteria, fungi and algae

→ Biocorrosion
Degaration by atmosphere

- physical: *abrasion* of constructions by particles (dust, sand) in wind

- aerosol = dust + water drops → visual damage
Degradation by atmosphere

Corrosion agents in atmosphere

\(\text{CO}_2\) natural occurrence in atmosphere 0.03 %
carbonic acid – the weakest acid, salts carbonates

\[\text{H}_2\text{O} + \text{CO}_2 \leftrightarrow \text{H}_2\text{CO}_3\]

1. carbonation of lime mortars (+) and concrete (-)

\[\text{Ca(OH)}_2 + \text{CO}_2 \leftrightarrow \text{CaCO}_3 + \text{H}_2\text{O}\]

mortar:       +  hardening
concrete:     -  reinforcement corrosion, disturbance of
              equilibrium composition of cement binder
Degradation by atmosphere

Corrosion agents in atmosphere

CO₂:

2. dissolution of carbonates
calcite \( \text{CaCO}_3 \): limestone, marble, marl, lime mortars

\[
\text{CaCO}_3 + \text{H}_2\text{O} + \text{CO}_2 \leftrightarrow \text{Ca}^{2+} + 2\text{HCO}_3^-
\]

- insoluble calcite
- water (rain, ground)
- soluble (1.6 g/l) calcium hydrogencarbonate
- reversible process \( \leftrightarrow \)

→ calcite dissolution (high CO₂ in water)

← calcite precipitation (low CO₂ in water, water evaporation)
Degradation by atmosphere

Corrosion agents in atmosphere

**Sulphur oxides** SO\(_2\) and SO\(_3\)

**Nitrogen oxides** NO\(_x\) (NO, NO\(_2\), N\(_2\)O\(_3\)....)

*origin*: industry, transport, energy production

**acid-forming oxides**: forms acid with water (rain, aerosol)

(acid rain): H\(_2\)SO\(_3\), H\(_2\)SO\(_4\), HNO\(_2\), HNO\(_3\)

\[
\text{Ca(OH)}_2 \text{ in concrete and mortars– is neutralized to salts}
\]

**carbonates** CO\(_3^{2-}\) – salts of carbonic acid (the weakest acid)

**are dissolved by other acids:**

\[
\text{CaCO}_3 + 2 \text{HNO}_3 \rightarrow \text{Ca(NO}_3)_2 + \text{CO}_2 + \text{H}_2\text{O}
\]

loss of strength, dissolution of limestone and marl – statues, historical buildings
Degradation by soluble salts

Common ions in pore solution in building materials:
Ca$^{2+}$, Na$^+$, NH$_4$$^+$
SO$_4$$^{2-}$, Cl$^-$, NO$_3$$^-$, CO$_3$$^{2-}$

Origin of salts in buildings:
- materials component (CaSO$_4$.2H$_2$O in gypsum)
- dissolved in ground water which attacks constructions
- products of corrosion by acid rains
- metabolic products of microorganisms and animals
- winter maintenance of roads and pavements
Degradation by soluble salts: mechanism

1. salt dissolves in water
   unsaturated solution (*unsaturated = water can dissolve even more salt*)
2. the unsaturated solution is transported by pore système (capillary elevation)
3. formation of saturated solution
   (*saturated – solution with the highest possible concentration of salt – e.g. NaCl 360 g/l*)
   →saturation due to **water evaporation** (summer)
4. further evaporation → **crystallization** of salt from saturated solution
Degradation by soluble salts: on surface

A) the saturated solution is formed on the surface of construction → crystals are visible - **efflorescence**

aesthetic problem – historical buildings, frescoes... efflorescence does not damage the construction
Degradation by soluble salts: crystallization in pore system

B) saturation is reached in pores of material (close to surface) → crystals are formed in the pores (subflorescence)
growing crystals generate crystallization pressure – crystals press on the pore walls → up to 100 MPa – higher than the material´s strength→
→ cracks in construction, flaking of plaster, disintegration of construction
Capillary elevation of salt solution in wall

Evaporation of salt solution in pore
Degradation by soluble salts: cyclic hydration and dehydration

**Hydrates:** crystals containing some molecules of water in structure

<table>
<thead>
<tr>
<th></th>
<th>Specific volume cm$^3$ g$^{-1}$</th>
<th>Expansion crystals volume cm$^3$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Na$_2$SO$_4$</td>
<td>0,373</td>
<td>0,373 (100 %)</td>
</tr>
<tr>
<td>Na$_2$SO$_4$·10H$_2$O</td>
<td>0,683</td>
<td>1,549 (415 %)</td>
</tr>
</tbody>
</table>

Hydration and dehydration depend on humidity of air: in winter are stable hydrates, in summer crystals without water. **Hydration pressure!**
Degradation by soluble salts: formation of insoluble salts

crystallization of \textbf{calcite} on the surface of concrete:

\[
\text{Ca(OH)}_2 + \text{CO}_2 \rightarrow \text{CaCO}_3 + \text{H}_2\text{O}
\]

- \text{solution of Ca(OH)}_2 in pores of concrete
- \text{from air}
- \text{insoluble calcite forms crystals on surface of concrete}
Biodegradation
Biodegradation: bacteria

- unicellular microorganisms
- both auto and heterothrophic
- wide range of environments

**sulphur and nitrifying bacteria:**
- are everywhere (water, soil, **cattle sheds**)
- normal temperature, feed, neutral pH...
- acquire energy by oxidation of S and N compounds
  → „produce“ sulphates $\text{SO}_4^{2-}$ and nitrates $\text{NO}_3^{-}$ →
- **source of soluble salts → salt degradation**

**prevention:** cleanliness, dry, light
Biodegradation:
rot and fungi

- heterotrophic organisms → acquire energy by oxidation of organic substances: **wood, dust, fabric, paper**
- environment: feed, damp, normal temperature
- damages: sap rot – damage wooden constructions
  mycelium fibers grow in walls – **mechanical damage**
  metabolic production of organic acids → dissolve calcite
- prevention: **fungicides**, dry, cleanness – no dust
mycelium

frUITING body
Biodegradation: algae

- autotrophic organisms → production of $\text{CO}_2$ → support
dissolution of calcite $\text{CaCO}_3$ (limestone, mortars)
- production of organic acid → dissolution of $\text{CaCO}_3$

- form colour slimy coatings – water structures
- algae growing in pores → expansion and mechanical damage
Biodegradation:

**Plants and Animals**

- **Mechanical damage:** growing roots, animals action in walls (insects, rats)

- **Animals excrements:** hosts bacteria → production of acids and salts → salt corrosion, dissolution of calcite
Biodegradation

- prevention of all kinds of biodegradation:
  - dry and light environment
  - cleanness
  - proper construction – insulation, water drainage
  - maintenance of building – reduction of ground water attack, rain in, presence of animals and plants...
Degradation of building materials
Degradation of building materials

- freezing-thawing and water action – affect all porous building materials
- chemical degradation – depends on chemical composition of the material
- biodegradation – wood is most sensitive, other materials are attacked by biodegradation in damp environment
Aggregates, stone-cutted products

- **aggregates made from magmatic rocks** (dominant in Czech Republic):
  - dense – no pores = no damage by ice and water
  - high chemical resistance

- **limestone aggregates**
  - calcite $\text{CaCO}_3$: attacked by acid rains and by $\text{CO}_2$ dissolution
Aggregates, stone-cutted products

- aggregates with high content of amorphous SiO$_2$ (opal, chalcedony) → causes Alkali-silica reaction - ASR

amorphous SiO$_2$ + NaOH (KOH)

→ gel of sodium silicate

→ absorbs water

→ expansion
Aggregates, stone-cutted products

- prevention of alkali-silica reaction
  1. no amorphous SiO$_2$ in aggregates
     maximum content – few percent
     highest content in river sediments (USA, China)
  2. reduce alkali content (Na$_2$O, K$_2$O) in cement
Aggregates, stone-cutted products

- **sandstone**
  - porous – sensitive to freezing-thawing and water action
  - usually contains some calcite $\text{CaCO}_3$ – attacked by acid rains and biocorrosion
Aggregates, stone-cutted products

- **crust on sandstone**: calcite in sandstone reacts with SO$_2$ – in polluted air (cities) →
  product is gypsum CaSO$_4$.2H$_2$O

- dark colour - dust
Aggregates, stone-cutted products

- **marlstone**: porous, high content of CaCO$_3$ → very sensitive to water, freezing-thawing, acid rains, biocorrosion → more suitable for interiors
Degradation of concrete
Physical degradation of concrete

- mechanical damage: flowing water, plants...
- freezing-thawing damage: depends on porosity
  higher water/cement ratio → higher porosity → higher freezing-thawing damage

- thermal degradation of concrete:
  1. decomposition of hydration products (CSH, CAH) (from 200°C)
  2. dehydration of Ca(OH)$_2$ (from 500°C)
     → loss of strength

- different thermal expansion of binder and aggregates: cracks on interface between binder and aggregates
Chemical degradation of concrete

1. **kind:** dissolution and leaching of binder
2. **kind:** chemical reactions of binder with environment resulting to **non-binding products** (frequently followed by efflorescence)
3. **kind:** chemical reactions with formation of **voluminous products → expansion**

All chemical corrosion processes cause loss of strength and cohesion

   Extreme cases: disintegration of concrete

4. **corrosion of steel reinforcement**
Concrete: 1. kind
dissolution and leaching

- dissolution of Ca(OH)$_2$ from cement binder in pore water and consequent **leaching out of Ca(OH)$_2$**

- dangerous water: „hungry water“ (soft) – with low content of Ca$^{2+}$ and other minerals (rain and river water)

- leaching is dangerous for water and underground structures

- prevention – proper waterproofing
Concrete: 1. kind

dissolution and leaching

- reduction of $\text{Ca(OH)}_2$ concentration in concrete causes disruption of equilibrium between components of cement binder → results to decomposition of CSH and CAH hydrates → decrease of strength
- decrease of $\text{pH}$ due to lower $\text{Ca(OH)}_2$ → damage of steel reinforcement
- calcite efflorescence is formed on the surface
Concrete: 2. kind
non-binding products

- **acid corrosion**: reaction of Ca(OH)$_2$ from cement binder with acid components of environment: acids of sulphur and nitrogen (H$_2$SO$_4$, H$_2$SO$_3$, HNO$_3$) from acid rain and biocorrosion
  - Ca$^{2+}$ salts are formed from Ca(OH)$_2$ – no binding ability
  - decrease of Ca(OH)$_2$ concentration results to disruption of equilibrium in cement binder and consequent decomposition of CSH and CAH binder
  - formed salt may be soluble → efflorescence

\[
\text{Ca(OH)}_2 + 2 \text{HNO}_3 \rightarrow \text{Ca(NO}_3)_2 + 2 \text{H}_2\text{O} \quad \text{efflorescence}
\]
\[
\text{Ca(NO}_3)_2 \cdot 2 - 4\text{H}_2\text{O}
\]
Concrete: 2. kind
non-binding products

- **carbonation of concrete:** reaction of Ca(OH)$_2$ from binder with CO$_2$ from air or water (water structures)

  - calcite CaCO$_3$ is formed

  \[
  \text{Ca(OH)}_2 + \text{CO}_2 \rightarrow \text{CaCO}_3 + \text{H}_2\text{O}
  \]

  - decrease of Ca(OH)$_2$ causes:
    - decrease of pH → **corrosion of steel reinforcement**
    - disruption of binder equilibrium
  - in extreme cases may start carbonation of CSH and CAH hydrates to SiO$_2$, Al$_2$O$_3$ → decrease of strength
Concrete: 2. kind

non-binding products

- Observation of concrete carbonation by phenolphthalein: colour change at pH 8-10

- No colour: pH ≈ 8
- Carbonated concrete no Ca(OH)$_2$
- Reinforcement is damaged

- Violet: pH > 10
- High Ca(OH)$_2$
- OK
Concrete: 3. kind
expansive products

- chemical reactions having **voluminous products** → are formed and crystallized in concrete and generated crystallization pressure – products expansion damages structure of concrete: decrease of strength, disintegration

![Diagram showing sulfate attack on grains boundaries and expansion of binder](image-url)
Concrete: 3. kind expansive products

- sulphate expansion (gypsum expansion)
sulphate attack on concrete: ground water
  sea water
  sulphates from aggregates

sulphides: may by found in aggregates, are spontaneously oxidized to sulphates

Test of aggregates on presence of sulphides and sulphates:
1. HCl decomposes sulphides to $H_2S$ (smells)
2. sulphates are precipitated by $BaCl_2$ as insoluble $BaSO_4$
Concrete: 3. kind expansive products

- sulphate expansion
  - mechanism:
    - 1. formation of gypsum

\[
\text{Ca(OH)}_2 + \text{SO}_4^{2-} + 2 \text{H}_2\text{O} \rightarrow \text{CaSO}_4 \cdot 2\text{H}_2\text{O} + 2\text{OH}^- \\
\]

formation of gypsum from SO\(_2\) from air:

\[
\text{Ca(OH)}_2 + \text{SO}_2 + \frac{1}{2}\text{O}_2 + \text{H}_2\text{O} \rightarrow \text{CaSO}_4 \cdot 2\text{H}_2\text{O} \\
\]
Concrete: 3. kind expansive products

- sulphate expansion
  - mechanism:

2. formation of ettringite

\[ 3\text{CaO}.\text{Al}_2\text{O}_3.6\text{H}_2\text{O} + 3(\text{CaSO}_4.2\text{H}_2\text{O}) + 19\text{H}_2\text{O} \rightarrow 3\text{CaO}.\text{Al}_2\text{O}_3.\text{CaSO}_4.32\text{H}_2\text{O} \]

hydrate C₃AH₆ from concrete
gypsum
ettringite expansion 2.65x
Concrete: 3. kind expansive products

- protection of concrete against sulphate expansion:
  - prevent from contact of concrete with high-sulphate water
  - sea structures:
    - cement with low C₃A (to 5 %)
    - low porosity of concrete (low w/c)
    - adding of pozzolana (slag) → it reacts with Ca(OH)₂ instead of gypsum formation
Concrete: 3. kind expansive products

- **magnesium expansion**

  1. **kind** reaction of soluble Mg$^{2+}$ salts (ground and sea water) with Ca(OH)$_2$ in concrete

  \[
  \text{MgSO}_4 + \text{Ca(OH)}_2 + 2 \text{H}_2\text{O} \rightarrow \text{Mg(OH)}_2 + \text{CaSO}_4 \cdot 2\text{H}_2\text{O}
  \]

  voluminous product

  2. **kind** reaction of MgO (v cementu) with water:

  \[
  \text{MgO} + \text{H}_2\text{O} \rightarrow \text{Mg(OH)}_2
  \]

  slow process: takes place in settled concrete
Concrete: salt corrosion

- **chloride corrosion**
  origin of chlorides: winter maintenance salt, sea water

CAH hydrates react with chlorides (NaCl, CaCl₂) to **Friedel's salt 3CaO.Al₂O₃.CaCl₂.10H₂O** (non-binding)

1. expansion of Friedel's salt
2. decrease of strength due to decrease of CAH concentration

- **salt corrosion**: efflorescence and crystallization pressure
Concrete: salt corrosion

- **sea water corrosion**: leaching out of Ca(OH)$_2$ and decomposition of CSH and CAH hydrates
  reaction of CAH to Friedel’s salt
  sulphate expansion
  magnesium expansion
  crystalization and cyclic hydration-dehydration of salts in pores of concrete

- **prevention of salt corrosion of concrete**
  low porosity (low w/c)
  low C$_3$A in concrete
  low Ca(OH)$_2$ in concrete – use of blended cements with pozzolana content
Concrete: corrosion of steel reinforcement

- fresh concrete: very high pH (alkaline ≈ 12) steel is **passivated** – layer of Fe(OH)$_3$ protects steel against corrosion

- carbonated concrete: pH slowly decreases → the passive layer is destroyed when pH decreases app. to 9,5 → corrosion of reinforcement takes place

- **loss of contact between concrete and reinforcement**
Concrete:
corrosion of steel reinforcement

- reinforcement protection
  - good concrete – no cracks – carbonation starts in cracks
  - stainless steel reinforcement – expensive
cathodic protection of reinforcement
Degradation of materials based on calcium carbonate CaCO₃

- Calcite CaCO₃ is dominant component of lime mortars and plasters, limestone, marlstone
- CaCO₃ is dissolved by all acids – both inorganic and organic (acid rains, biodegradation)

CaCO₃ + 2 HNO₃ → Ca(NO₃)₂ + CO₂ + H₂O

CaCO₃ + H₂SO₃ + \( \frac{1}{2} \) O₂ + H₂O → CaSO₄.2H₂O + CO₂

- Mortars with cement – higher corrosion resistance
Degradation of gypsum

- gypsum = typical air binder
- solubility of CaSO₄·2H₂O: 2.4 g/l
- dissolves mainly in flowing water
- solution of CaSO₄·2H₂O is acid → steel corrosion
  gypsum can not be reinforced
- gypsum in wet environment: hydrofobization
  (hydrofobobized gypsum repels water)
- thermal stability of gypsum: very low
  gypsum decomposes from 60 °C, from 120 °C
  decomposes rapidly to hemihydrate CaSO₄·1/2 H₂O
Degradation of ceramics

- ceramics: high resistance to thermal and chemical action (dissolves only in HF)
- physical corrosion: degradation by freezing-thawing action and by \textbf{salts}

- resistance depends on granulometry of raw materials (Winkler diagram)

- unfired bricks: sensitive to flood
Fort Jefferson, Florida
Degradation of glass

- weak point - brittleness
- chemical corrosion: glass dissolves in HF and in highly alkaline solutions (cheap or old glass in dishwasher)
- increase of resistivity to alkalies: boiling of glass in water or acid – surface layer in enriched by SiO₂ – more resistant to alkalies
Wood degradation

- biodegradation – rot, insects
- oscillation of temperature and humidity: causes contraction and swelling → cracks
- chemical degradation: wood is attacked by oxidising agents, acids and bases → depolymeration and oxidation of cellulose, change of color and mechanical properties
- wood of coniferous trees is more chemically stable than wood of deciduous
- wood protection: fungicides, biocides, fire protection
Degradation of synthetic polymers

- depends on composition of the polymer
- generally: high chemical stability
  
  **low thermal stability**: softening, melting, burning, thermal decomposition

  **UV radiation** (sun) causes **photochemical reactions** in polymer – decomposition of polymer chain (embrittlement)

- biocorrosion of polymers: organic compounds serve as nutrient for fungi (rot) and bacteria
Goals

- describe principals of physical corrosion of building materials
- degradation by water action
- degradation by aggressive gasses
- behaviour of salt in porous materials
- biodegradation
- chemical degradation of concrete
6B. Analytical chemistry
Analytical chemistry in civil engineering

- **analysis of materials**
  composition of cement, lime etc., content of sulphates in aggregates

- **environmental analysis**
  composition of water: drinking, mixing water for concrete, ground water – contains dangerous components for building materials
  composition of soil: contamination by toxics

- **waste analysis before landfilling**
  rubble composition, contaminated soils (oil, heavy metals Hg, Cu, Pb, Cd...)
Sampling

- **representative sample** contains all components of the material in the same ratio as are present in the sampled material.

- Sampling of liquids and gasses: usually homogeny – no problem.

- Sampling of solids: the material is crushed, homogenized, quartered.

- **Sampler for solids and powders**

- **Quartering**
Chemical analysis

- qualitative: composition of sample (elements, ions, molecules)

- quantitative: determination of concentration of components

- conventional: **volumetry** and **gravimetry**

- instrumental: by help of instruments
Volumetry - titration

- for determination of concentration of a compound in liquid solution
- unknown solution = **analyte**
- **reagent** = standard solution of a compound (titrant)
- principle: analyte reacts with **equivalent** amount of reagent according known chemical reaction
- endpoint of titration – by visual **indicator** (point of equivalence)
Volumetry - titration

- **acid-base titration:**
  - for determination of acid concentration by help of KOH reagent
  - acid reacts with a base
  - endpoint indicated by pH measurement (colour change of a chemical indicator, pH meter)

\[
\text{analyte} \quad \text{reagent}
\]

\[
\text{H}_2\text{SO}_4 + 2 \text{KOH} \leftrightarrow \text{K}_2\text{SO}_4 + 2 \text{H}_2\text{O}
\]
Volumetry - titration

\[ \text{H}_2\text{SO}_4 + 2 \text{KOH} \leftrightarrow \text{K}_2\text{SO}_4 + 2 \text{H}_2\text{O} \]

\[ \frac{n_{\text{ACID}}}{n_{\text{KOH}}} = \frac{1}{2} \]

\[ n_{\text{ACID}} = \frac{1}{2} \cdot n_{\text{KOH}} \]

\[ c_{\text{ACID}} \cdot V_{\text{ACID}} = \frac{1}{2} \cdot c_{\text{KOH}} \cdot V_{\text{KOH}} \]

\[ c_{\text{ACID}} = \frac{1}{2} \cdot \frac{c_{\text{KOH}} \cdot V_{\text{KOH}}}{V_{\text{ACID}}} \]
Gravimetry

- Analyte is precipitated by a chemical reaction in the form of a known and insoluble product.
- Fe$^{3+}$ ions are precipitated in alkaline environment as insoluble Fe(OH)$_3$.

Fe$^{3+}$ + 3 OH$^-$ → Fe(OH)$_3$

$$C_{Fe} = \frac{n_{Fe}}{V} = \frac{m_{Fe(OH)3}}{M_{Fe(OH)3}}$$

filtration, drying, weighing