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PRINCIPLE OF THE FORMATION OF EFFLORESCENCE ON THE SURFACE OF AUTOCLAVED POROUS CONCRETE PRODUCTS

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If for the production of autoclaved porous concrete materials is employed as the main raw component quartz sand with a considerable content of sodium, potassium or sodium-potassium feldspars, under suitable conditions in the atmosphere efflorescence are set up in the civil engineering structure after their application. Their essence is thenardite (Na_2SO_4), the formation of which is caused by the decomposition of sodium feldspar with $Ca(OH)_2$ in the course of the autoclave process and through the subsequent reaction with the present sulphate ions. The degree of decompositon of potassium feldspar is under the same conditions pronouncedly lower. With regard to the lowest solubility of thenardite in water in comparison with the other alkalic sulphates there occurs the undesirable formation of efflorescence not only on edges, but also on the surfaces of porous concrete products.

INTRODUCTION

For the manufacture of autoclaved building materials is used beside power station fly-ash, blast furnace slag and further secondary raw materials, apart from CaO and cement, in a pronounced measure quartz sand with various degrees of purity.

The main aim of correctly managed hydrothermal processes is the formation of certain calcium hydrosilicate components which are characterized, with regard to a suitable morphology of their particles, by pronounced bonding properties [1–4]. Beside hydrosilicate and hydrogarnet components can be formed under certain conditions hydroxylellestadite [$Ca_{10}(SiO_4)_3(SO_4)_3(OH)_2$], abbreviated notation HXEL which exhibits no bonding affects [5]. In case of the smallest particles of quartz grains ($< 10 \mu m$) is formed the above mentioned phase only in the initial period of the hydrothermal process, after that it is decomposed with the formation of tobermorite and $CaSO_4$(III). The decomposition of HXEL is shifted in time in proportion to the increasing granulometry of the quartz [6–8].

In connection with the formation of undesirable efflorescence on blocks and elements from porous concrete manufactured from quarried quartz sand was highly desirable to prove the cause of their formation and to try, if possible, to reduce their appearance or even to prevent their formation. Through experimental work has been completely eliminated the possibility of efflorescence formation from the employed cement and lime or power station fly-ash as the individual raw-materials components, including the used water.

For these reasons further work was aimed onto quartz sand, quarried in the vicinity of the works (HR) for the manufacture of porous concretes.

The sand contained a pronounced proportion of feldspars, the decomposition of which in the course of the autoclave process, especially in the presence of lime, has been unambiguously proved [9–10]. On the basis of these experiences was employed quartz sand, used for the production of porous concretes, in which efflorescence have been found and furthermore a quartz raw materials, employed for the manufacture of porous concretes by an identical technology, in which no formation of efflorescence has been observed.

By X-ray measurement methods was further proved that the formed efflorescence are created by thenardite, anhydrous sodium sulphate (Na_2SO_4). For this reason experimental work was aimed at the delimitation of the decomposition of feldspars contained in quartz sand.

EXPERIMENTAL

Employed raw materials and method of their processing

For the work itself were employed quartz sands and raw mixes from porous concrete production plants (HR) and (CHL). In the first plant is being processed a quartz raw material which contains a considerable proportion of feldspars, in the second their contents

Table I

Results of the chemical analyses of the employed materials

Determined components (%) — Identification of employed materials	Quartz sand (HR)	Quartz sand (CHL)	Raw material mixture (HR)	Raw material mixture (CHL)	Cement (MA)
SiO_2	74.90	93.50	50.57	61.99	21.36$^+$
Al_2O_3	11.69	2.90	8.17	2.37	5.34
Fe_2O_3	2.07	0.17	1.65	0.76	3.42
TiO_2	0.34	0.08	0.28	0.22	0.26
CaO	1.76	1.35	29.85	28.95	65.01
MgO	1.06	0.65	1.02	0.92	2.18
K_2O	5.00	0.94	3.27	0.51	0.90
Na_2O	2.10	0.11	1.17	0.14	0.29
SO_3	0.12	0.01	0.48	0.53	0.07
ignition loss	1.40	0.65	3.72	3.58	1.00
Total	100.44	100.36	100.18	99.97	99.83

$^+$ SiO_2 + undecomposed fraction

are considerably lower. Both plants use the same technological system and the composition of the raw mix corresponds to 62 p. quartz sand, 20 p. portland cement and 18 p. lime. The results of the chemical analysis of the basic raw materials are presented in Table I.

On the basis the results of X-ray diffraction analyses the quartz sand (HR) contains beside a predominant quantity of β-quartz a significant proportion of feldspars and kaolinite, whereas the sand (CHL) is characterized by a high content of β-quartz and a very low quantity of feldspars and kaolinite.

Beside clinker (MA) is used power station fly-ash (NO) which has the following chemical composition (%): moisture: 0.30; ignition loss: 2.29; SiO_2: 59.73; Al_2O_3: 17.34; Fe_2O_3: 8.99; TiO_2: 0.83; CaO: 6.43; MgO: 2.09; K_2O: 1.50; Na_2O: 0.58; SO_3: 0.17.

Method of processing the specimens

From both raw mixes have been weighed 3.200 g of specimen and transferred quantitatively into small pressure vessels; 30 ml boiled distilled water was added and into each vessel 6 pcs steel wire (length 10 mm, thickness 3 mm). The small pressure vessels were mounted in the rotation device of a hot-air cabinet, so that during the whole period of hydrothermal processing (193°C, 5 h) they were slowly rotating. After the termination of the process the suspensions were filtered, the filter sediment was washed many times with hot distilled water, after cooling the filtrate was topped up to a volume of 500 ml in a volumetric flask. The residue on the filter was dried at 105°C in an N_2 stream. The results of the chemical analyses of the starting raw mixes, the residues after leaching and filtration are presented in the Tables II and III.

From the results in the case of the two raw mixes can readily be seen that a substantially higher degree of decomposition was determined in the case of sodium feldspar in comparison with potassium feldspar.

Decomposition of feldspars in quartz sand (HR)

With regard to the significant contents of alkalis in the quartz sand (HR) which is used as the main raw-material component for the production of porous concrete in the plant (HR), it was desirable to determine the transition of alkalis into the solution upon employing a mixture of quartz sand with CaO without gypsum rock or with a 3% admixture of the latter. From the compounded mixtures suspensions have been preparad (solid phase : $H_2O = 1 : 10$) which have been processed in small pressure vessels at 193°C for a period of 1 and 5 h (isothermal holding period).

The composition of the mixtures and the results of the degree of decomposed feldspars are shown in Table IV.

The quantity of alkalis was converted to albite ($NaAlSi_3O_8$) and orthoclase ($KAlSi_3O_8$). The con-

Table II

Raw material mixture (HR)

Determined components (%) Identification of the specimen	Original$^+$ raw material mixture	Residue after leaching	Filtrate^{++}
K_2O	3.40	2.89	15.00
Na_2O	1.22	0.45	63.11
SO_3	0.50	0.20	60.00
CaO	31.00	30.67	1.06

Table III

Raw material mixture (CHL)

Determined components (%) Identification of the specimen	Original$^+$ raw material mixture	Residue after leaching	Filtrate^{++}
K_2O	0.53	0.45	15.09
Na_2O	0.14	0.05	64.28
SO_3	0.55	0.24	56.36
CaO	30.02	29.22	2.66

$^+$ Converted to annealed fraction

$^{++}$ Components in the filtrate, expressed in % of their contents in the original mixture

version is not completely exact, since in the employed sand can be present in a very low content further minerals, e.g. nepheline and others which contain a certain quantity of alkalis ($K_2O + Na_2O$).

As can readily be seen from the results, after a 1 hour hydrothermal process have been decomposed only 16.12% of the sodium and 8.66% of the potassium feldspar. In the mixture with gypsum rock the proportion of soluble alkalis has increased, so that the quantity of Na-feldspar corresponds to 19.59% and of K-feldspar to 9.16%.

It is not without interest that by prolonging the autoclave process the quantity of decomposed sodium feldspar in the presence of gypsum rock increased to 52.46%, whereas the proportion of decomposed potassium feldspar corresponds only to 17.41%.

Determination of the degree of decomposition of feldspar specimens under hydrothermal conditions

With regard to the previous results it was desirable to carry out experimental work with feldspar specimens. For this purpose have been obtained from the mineralogical department of the Moravian Museum in Brno specimens of orthoclase (I), orthoclase (II), albite and oligoclase.

Even though these were not pure, representative minerals, as can readily be seen from the results of a sectional chemical analysis (Table V), they are due to their composition and nature suitable for the study of hydrothermal reactions.

The individual starting components were after rubbing and screening on the No. 325 sieve homogenized with cement (MA) and CaO (analyt. grade). The composition of the mixtures was selected in such a way as to approach practical production conditions.

The suspensions of the individual mixtures were hydrothermally processed in small pressure vessels at 193°C, isothermal holding period 5 h. The starting compositions of the mixtures and the results of the chemical determination are presented in Table VI.

From the results is apparent a markedly higher de-

Table IV

Degree of decomposition of albite and orthoclase in a mixture of quartz sand (HR) with CaO and $CaSO_4.2H_2O$

193°C / 1 h

Identification of the mixture	Composition of the mixture	Degree of decomposition of Na feldspar (%)	Degree of decomposition of K feldspar (%)
1A	2.40 g quartz sand (HR) 0.80 g CaO 30 ml H_2O	16.12	8.66
2A	2.40 g quartz sand (HR) 0.80 g CaO 0.096 g $CaSO_4.2H_2O$ 30 ml H_2O	19.59	9.16
193°C / 5 h			
1B	2.40 g quartz sand (HR) 0.80 g CaO 30 ml H_2O	40.92	11.53
2B	2.40 g quartz sand (HR) 0.80 g CaO 0.096 g $CaSO_4.2H_2O$ 30 ml H_2O	52.46	17.41

Table V

Results of a sectional chemical analysis

Identification of the specimen	Determined components (%)	CaO	Na_2O	K_2O
orthoclase (I)		0.31	3.52	10.89
orthoclase (II)		0.44	1.99	12.44
albite		0.65	7.27	1.99
oligoclase		0.47	4.99	8.61

gree of decomposition of sodium feldspar in comparison with potassium feldspar.

By further experiments with graded additions of $CaSO_4.2H_2O$ was unambiguously proved that a different gypsum rock content exhibits no more substantial influence onto the decomposition of sodium or potassium feldspar.

Delimitation of the influence of feldspar onto the physical and mechanical properties of dense autoclaved mixtures

With regard to the pronounced proportion of feldspars in quartz sands used as one of the main raw material components for production of autoclaved building materials, it was essential to delimitate their principial influence above all onto the attained strengths.

For the actual experiments was employed quartz sand (CHL) which is characterized by a low proportion of alkalis. We used potassium feldspar (orthoclase II) and further components, as can be seen from Table VII.

Table VI

Degree of decomposition of feldspar specimens in a mixture with cement and CaO

Identification of the mixture	Composition of the mixture	Degree of decomposition of Na feldspar (%)	Degree of decomposition of K feldspar (%)
1	1.984 g orthoclase (I) 0.640 g cement (MA) 0.576 g CaO(analyt. grade) 30 ml H_2O	57.46	30.00
2	1.984 g orthoclase (II) 0.640 g cement (MA) 0.576 g CaO(analyt. grade) 30 ml H_2O	59.98	29.10
3	1.984 g albite 0.640 g cement (MA) 0.576 g CaO(analyt. grade) 30 ml H_2O	48.62	17.14
4	1.984 g oligoclase 0.640 g cement (MA) 0.576 g CaO(analyt. grade) 30 ml H_2O	54.42	29.01

Table VII

Composition of the mixtures and compression strength values

Identification of the mixture	Orthoclase (II)		Quartz sand (CHL) (g)	Cement (MA) (g)	CaO (g)	H_2O (ml)	Compressive strength (MPa)
	(g)	(%)					
1	–	–	40.300	13.00	11.70	25.0	69.5
2	0.806	2	39.494	13.00	11.70	25.0	66.7
3	2.015	5	38.285	13.00	11.70	25.0	63.1
4	4.030	10	36.270	13.00	11.70	25.0	62.8
5	8.060	20	32.240	13.00	11.70	25.0	59.9
6	12.100	30	28.200	13.00	11.70	25.0	54.1

Quartz sand and feldspar were after grinding ($< 60\mu m$) mixed in such a way that orthoclase was added at the expense of quartz, so that quartz sand : feldspar = 100:0; 98:2; 95:5; 90:10; 80:20 and 70:30. After the addition of further components and thorough homogenization wetted mixtures were prepared which were hydrothermally processed (193°C / 5h) in the from of microcubes with a 20 mm edge.

It follows from the compressive strength values that with an increasing proportion of feldspar in the employed quartz sand takes place a decrease of the strengths, proportional to the feldspar fraction.

Experiments carried out with sodium feldspar (albite) have shown an analogical phenomenon which manifests itself in a certain reduction of compressive strengths.

Possibilities of preventing efflorescence on the surface of autoclaved porous concrete products

As long as is employed as the main raw material component for the production of porous concretes quartz sand with a proportion of feldspars, under hydrothermal conditions they are decomposed through the action of $Ca(OH)_2$ and sulphates are formed by reaction with the present SO_4-ions. Highly undesirable is the formation of thenardite (Na_2SO_4) which crystalizes in the orthorhombic system. Its solubility in water is with regard to the other alkalic sulphates relatively low, since it corresponds to 4.76 g Na_2SO_4/100 ml H_2O at 20°C.

In the case of monoclinic Na_2SO_4 its solubility is practically unlimited, the solubility of rhombic $Na_2SO_4.7H_2O$ represents 19.5 g/100 ml H_2O and of monoclinic $Na_2SO_4.10H_2O$ even 58.3 g/100 ml H_2O.

It follows from the above data that the solubility of thenardite is significantly lower in comparison with the other alkalic sulphates – in the case of K_2SO_4 it represents 12.3 g/100 ml H_2O.

This fact causes that under suitable atmospheric conditions (relative humidity and temperature of the atmosphere) thenardite is separated from the solution which gradually diffuses from the inside parts of the porous concrete mass to its surface.

In order to verify the above phenomenon there have been prepared from a current production block with the utilization of quartz sand (CHL), characterized by a low proportion of feldspars, two porous concrete specimens with the dimensions 10 × 10 × 15 cm. One of them was dipped into a 2.5% Na_2SO_4 solution, the other into a 2.5% K_2SO_4 solution. Both solutions with the porous concrete specimens were maintained for 2 h at a mild boil and after cooling their surface was thoroughly washed with distilled H_2O. Then from a distance of approx. 1 m the specimens were blown by air from an electric fan for a period of approx. 4 h to attain an increased rate of migration of the solution (moisture) in the porous concrete mass in the first time period of the experiment.

Already after 16 h has formed on the block in the Na_2SO_4 solution a thin, in places continuous white coating, the thickness of which increased in proportion with time. After 1 week of storage the above specimen was coated by an almost continuous layer of efflorescence which especially on the edges attained a thickness of up to 7 mm. Their further growth – the attained thickness of the efflorescence – was limited by the relatively low cohesion of the formed crystals which were falling of and accumulated on the support plate. It was proved by an X-ray diffraction analysis that the efflorescence on the porous concrete specimen represent thenardite (Na_2SO_4).

But the porous concrete specimen after application of the K_2SO_4 solution did not show even an indication of the formation of efflorescence, so that not only the surface of the block, but also its edges were absolutely clean.

The preliminary results have confirmed the assumption that for the prevention of undesirable efflorescence it is expedient to reduce the calcium hydroxide concentration during the first reaction period to an acceptable limit.

In order to verify this assumption was used quartz dust of an X-ray amorphous nature, diatomaceous earth, blast furnace slag and further materials. With regard to the economic availability was for the actual experiments selected power station fly ash (NO), the addition of which amounted to 10–30–50 and 70% at the expense of clinker (MA). The mixtures were hydrothermally processed in an identical manner as the previous ones.

The compositions of the mixtures and the results of the analyzed extract are presented in Table VIII.

Even though the SO_3 content in the extract fluctuates somewhat in a certain concentration range, from the Na_2O results follows unambiguously that with an increasing proportion of power station fly-ash is reduced the quantity of the given component in the filtrate. At the same time it is necessary to take into account the fact that the alkalis are simultaneously leached out from the present portland cement clinker and the power station fly-ash, to a slight degree also from gypsum rock.

As long as in mixture (6) the sand (HR) was replaced with pure quartz (analyt. grade), in the filtrate were determined 3.50 mg Na_2O/500 ml and 4.12 mg K_2O/500 ml. If these quantities are subtracted from the data for alkalic oxides in the case of mixture (6), then can readily be seen that from the sand (HR) go over into the solution only 9.41 mg Na_2O/500 ml and 3.86 mg K_2O/500 ml.

The prepared porous concrete specimens, in which the cement was partly replaced with power station fly-ash (cement : power station fly-ash = 4 : 1), were subjected to a test to determine their susceptibility to the formation of efflorescence. Whereas on porous concrete blocks without the addition of fly-ash efflorescence were formed not only on the edges, but almost on the whole surface, on the specimen with the addition of power station fly-ash practically no efflorescence were formed. It follows unambiguously from the above that for the suppression of the formation

Table VIII

Composition of the mixtures and results of the chemical determination

Ident. of mixture	Quartz sand (HR)		CaO (anal. grade)		Clinker (MA)		Power station fly-ash (NO)		$CaSO_4.2H_2O$		H_2O	SO_3	Na_2O	K_2O
	(g)	(%)	(g)	(%)	(g)	(%)	(g)	(%)	(g)	(%)	(ml)	*	*	*
1	1.984	62.00	0.576	18.00	0.600	18.75	–	–	0.040	1.25	30	15.43	25.61	7.49
2	1.984	62.00	0.576	18.00	0.540	16.88	0.060	1.87	0.040	1.25	30	16.63	24.12	6.54
3	1.984	62.00	0.576	18.00	0.420	13.13	0.180	5.62	0.040	1.25	30	14.74	22.13	7.21
4	1.984	62.00	0.576	18.00	0.300	9.38	0.300	9.37	0.040	1.25	30	18.34	19.53	7.40
5	1.984	62.00	0.576	18.00	0.180	5.63	0.420	13.12	0.040	1.25	30	16.81	17.15	7.80
6	1.984	62.00	0.576	18.00	–	–	0.600	18.75	0.040	1.25	30	18.35	12.91	7.98

* mg/500ml

of efflorescence which are represented by thenardite, it is expedient to use a mixed cement on the basis of power station fly-ash.

CONCLUSION

There was studied the cause of bloom formation on both blocks and panels made from autoclaved porous concrete upon the utilization of quartz sand as one of the main raw material components. It was proved by X-ray diffraction analysis that the efflorescence represent anhydrous sodium sulphate: thenardite (Na_2SO_4). For the experiment was used quartz sand and a raw mix from a plant, in which efflorescence appear frequently on the products, beside analogical raw materials from another plant where the products exhibit no defects. It was proved that the quartz sand from the first plant exhibits a several times higher content of alkalis in the from of feldspars in comparison with the raw material from the second plant.

The quartz sand specimens have been subjected to the hydrothermal process with the addition of water and CaO and it was found that the sodium feldspar decomposes in the course of the hydrothermal process to a substantially greater degree in comparison with potassium feldspar. This fact was also verified on representative specimens of relatively pure feldspare. With regard to the relatively low solubility of thenardite in water there occurs the formation of udesirable efflorescence.

For these reasons is recommended a mixed cement as one of the raw material components which contains power station fly-ash instead of pure portland cement. It has thus been possible in the optimal case to prevent the formation of efflorescence or at least to reduce their appearance onto a tolerable measure.

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PRINCIP VZNIKU VÝKVĚTŮ NA POVRCHU AUTOKLÁVOVANÝCH PÓROBETONOVÝCH VÝROBKŮ

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K výrobě autoklávovaných pórovitých betonů a dalších vápenopískových produktů je v některých závodech použito křemičitého písku, který obsahuje sodné, draselné nebo sodnodraselné živce, posléze způsobující tvorbu nežádoucích výkvětů na vyrobených tvárnicích nebo velkorozměrných dílcích, příp. na jiných výrobcích.

Rentgenometrickým stanovením bylo zjištěno, že vzniklé výkvěty na uvedených výrobcích představují thenardit (Na_2SO_4) a to zřejmě v souvislosti s jeho nejnižší rozpustností ve vodě v porovnání s ostatními sírany.

Hlavním cílem práce je zjištění principu jejich vzniku při užití křemičitého písku používaného v závodě, ve kterém na výrobcích dochází k tomuto nežádoucímu jevu a to ve výrazné míře. Bylo prokázáno, že v použitém křemičitém písku je obsaženo 5,0% K_2O a 2,1% Na_2O. K porovnání použito křemičitého písku ze závodu, na jehož výrobcích se výkvěty nevyskytují. Jak se dalo předpokládat, obsah alkálií je výrazně nižší (K_2O : 0,94%; Na_2O : 0,11%). Vedle uvedených křemičitých surovin ještě použito vzorku surovinové směsi z obou závodů na výrobu pórovitých betonů.

Bylo prokázáno, že ve směsi křemičitého písku s vápnem a sádrovcem v podobě suspenze stupeň rozkladu obsaženého Na-živce v průběhu autoklávového procesu je výrazně vyšší v porovnání s K-živcem. Tato skutečnost byla zcela jednoznačně potvrzena při užití vzorků poměrně čistých živců.

Konečně byly provedeny pokusy, v nichž cement byl částečně nahrazen elektrárenským popílkem a prokázáno, že výskyt výkvětů byl téměř zcela eliminován.

Na základě dosažených výsledků lze předpokládat, že užitím směsných cementů na bázi elektrárenského popílku bude možno tvorbu výkvětů na povrchu autoklávovaných výrobků snížit na únosnou míru nebo jejich vzniku zcela zamezit.

Výročí

Dne 19. 7. 1992 se dožívá 70 let Prof. Ing. Dr. Vladimír Šatava DrSc., významný vědecký pracovník v oboru chemie silikátů, vynikající vysokoškolský učitel a spoluzakladatel tohoto časopisu.

Po studiu a maturitě na reálce J. Nerudy v Praze absolvoval abiturientský kurs chemie a byl zaměstnán jako chemik. Po skončení války vystudoval Vysokou školu chemicko-technologického inženýrství ČVUT (nyní VŠCHT) v Praze a v r. 1948 nastoupil na místo asistenta Ústavu sklářství a keramiky, kde působil až do odchodu do důchodu v r. 1991, a kde působí jako učitel postgraduálního studia dodnes. Habilitoval se v r. 1962, vědeckou hodnost DrSc. získal v r. 1968, řádným profesorem byl jmenován v r. 1983. V letech 1962–1965 byl proděkanem fakulty chemické technologie, v letech 1962–1974 byl vedoucím Společné laboratoře pro chemii a technologii silikátů ČSAV a VŠCHT, stál u jejího zrodu a formuloval koncepci její činnosti. V letech 1957–1967 zastával funkci prvního vedoucího redaktora tehdy nově založeného časopisu Silikáty.

Po celou dobu svého působení na VŠCHT dělil svůj čas mezi práci pedagogickou a vědeckou. Díky jeho koncepčnímu přístupu se podařilo postupně přeměnit původní empirickou, tradiční disciplínu na teoreticky fundovaný obor, který je významnou součástí nově konstituovaného směru materiálového inženýrství na VŠCHT. Osobnost Prof. Šatavy byla po dlouhá léta zárukou a měřítkem úrovně a kvality pedagogické i vědecké práce na ústavu, přispívala ke vzniku náročného prostředí, nastavovala vysokou laťku způsobilosti k učitelské a vědecké práci. Takto působí Prof. Šatava i dnes svou nadále pokračující spoluprací s ústavem a prostřednictvím stále používané řady skript pro teoretické předměty oboru. Prof. Šatava vytvořil to, co se označuje jako „škola“ ve smyslu vyhraněné skupiny jím vychovaných tvůrčích pracovníků, kteří se kolem něj soustřeďovali, a kteří se k němu i po letech hlásí. Cit pro talentované mladé lidi a jejich výchova k zaujetí pro vědu a techniku, k exaktnímu myšlení a k originální tvůrčí práci, to jsou charakteristické rysy Šatavovy školy. Náročnost, ale přitom vlídný osobní vztah Prof. Šatavy ke studentům, vytvářely nezapomenutelné prostředí a trvalý pocit sounáležitosti bývalých studentů se školou a učitelem. To je nejvyšší ocenění úspěšné činnosti vysokoškolského učitele, jehož se Prof. Šatavovi zaslouženě dostává.

Přejeme jubilantovi do dalších let dobré zdraví a těšíme se na další spolupráci.

Redakce