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SYNTHESIS OF A SODIUM POLYPHOSPHATE COMPOSITION FROM ORTHOPHOSPHORIC ACID AND SODIUM SILICATE AND INVESTIGATION OF ITS THERMAL PROPERTIES

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Annotatsiya. Maqolada ortofosfat kislotasi (85 %), natriy gidroksid va natriy silikat asosida bir bosqichli, past haroratli usulda natriy polifosfat–silikat kompozitsiyasini sintez qilish natijalari keltirilgan. Reaksiya aralashmasining hisoblangan mol nisbatlari $Na/P = 1,62$ va $Si/P = 0,38$ ni tashkil etadi. 120–150 °C da quritilgan va 300 °C da 1 soat polikondensatlangan mahsulotning termik xossalari argon oqimida (100 ml/min), 10 °C/min tezlikda DTG-60 derivatografida o‘rganildi. TG egri chizig‘ida 775 °C gacha to‘rtta massa yo‘qotish bosqichi qayd etildi, umumiy massa yo‘qotish 7,97 % ni tashkil qildi. 289–375 °C oralig‘idagi 3,61 % massa yo‘qotish qoldiq P–OH guruhlarining polikondensatsiyasiga to‘g‘ri keladi va 300 °C dagi issiqlik bilan ishlov berish nazariy to‘liq kondensatsiyaning taxminan 76 % ini ta‘minlaganini ko‘rsatadi. 627,9 °C dagi keskin endotermik effekt massa o‘zgarishsiz kechadi va $NaPO_3$ ning adabiyotdagi suyuqlanish haroratiga (628 °C) to‘liq mos keladi; bu mahsulotda zanjirsimon polifosfat fazasi hosil bo‘lganini tasdiqlaydi. Olingan mahsulot argon muhitida 775 °C gacha termik barqaror.

Kalit so‘zlar: natriy polifosfat, metafosfat, polikondensatsiya, natriy silikat, silikofosfat, termogravimetrik tahlil, DTA, DTG, termik barqarorlik.

Аннотация. В статье приведены результаты одностадийного низкотемпературного синтеза полифосфатно-силикатной композиции на основе ортофосфорной кислоты (85 %), гидроксида натрия и силиката натрия. Расчётные

мольные соотношения реакционной смеси составляют $\text{Na/P} = 1,62$ и $\text{Si/P} = 0,38$. Термические свойства продукта, высушенного при 120–150 °C и поликонденсированного при 300 °C в течение 1 ч, изучены на дериватографе DTG-60 в токе аргона (100 мл/мин) при скорости нагрева 10 °C/мин. На кривой ТГ до 775 °C зафиксированы четыре стадии потери массы с суммарной потерей 7,97 %. Потеря массы 3,61 % в интервале 289–375 °C соответствует поликонденсации остаточных Р–ОН групп и показывает, что термообработка при 300 °C обеспечивает лишь около 76 % теоретически полной конденсации. Резкий эндотермический эффект при 627,9 °C протекает без изменения массы и полностью совпадает с литературной температурой плавления NaPO_3 (628 °C), что подтверждает образование цепочечной полифосфатной фазы. Полученный продукт термически устойчив в атмосфере аргона до 775 °C.

Ключевые слова: полифосфат натрия, метафосфат, поликонденсация, силикат натрия, силикофосфат, термогравиметрический анализ, ДТА, ДТГ, термическая устойчивость.

Abstract. This paper reports a one-pot, low-temperature synthesis of a sodium polyphosphate–silicate composition from orthophosphoric acid (85 %), sodium hydroxide, and sodium silicate. The calculated molar ratios of the reaction mixture are $\text{Na/P} = 1.62$ and $\text{Si/P} = 0.38$. The thermal behaviour of the product, dried at 120–150 °C and polycondensed at 300 °C for 1 h, was studied on a DTG-60 thermal analyser under flowing argon (100 mL/min) at a heating rate of 10 °C/min. Four mass-loss steps were recorded on the TG curve up to 775 °C, with a total mass loss of 7.97 %. The 3.61% loss over 289–375 °C corresponds to polycondensation of residual P–OH groups and indicates that heat treatment at 300 °C achieves only about 76% of theoretically complete condensation. The sharp endothermic effect at 627.9 °C proceeds without any change in mass and coincides with the melting point reported in the literature for NaPO_3 (628 °C), confirming the formation of a chain polyphosphate phase. The product is thermally stable in argon up to 775 °C.

Keywords: sodium polyphosphate, metaphosphate, polycondensation, sodium silicate, silicophosphate, thermogravimetric analysis, DTA, DTG, thermal stability.

Introduction. Condensed phosphates, and in particular chain sodium polyphosphates of the general formula $(\text{NaPO}_3)_n$, are used in many branches of modern chemical technology. Owing to their ability to bind Ca^{2+} and Mg^{2+} ions (sequestration), they are employed as scale inhibitors in water-treatment systems, as complexing builders in detergents, in the food industry, and as flotation reagents in mineral processing [1, 2].

Blended inhibitors containing both phosphate and silicate components are of particular practical interest for the simultaneous control of corrosion and deposit formation in drinking-water distribution networks: silicate ions form a protective film on the metal surface, whereas polyphosphate chains form soluble complexes that retain iron and hardness salts in solution [3, 4]. Compositions in which the polyphosphate and silicate components are combined within a single synthesis are therefore a promising direction of research [5].

The conventional route to sodium polyphosphates is the thermal dehydration of sodium dihydrogen orthophosphate in the range 600–900 °C followed by rapid quenching of

the melt to yield a glassy product. This route is energy-intensive and requires dedicated melting equipment. The development of methods that allow polycondensation to be performed at comparatively low temperatures (300–400 °C) is therefore a relevant task [6, 7].

The present work aimed to synthesise a sodium polyphosphate–silicate composition by a one-pot, low-temperature route from orthophosphoric acid, sodium hydroxide, and sodium silicate, and to study the dehydration and polycondensation processes of the resulting product by thermogravimetry (TG) and differential thermal analysis (DTA) [8, 9].

Objects and methods of research

Orthophosphoric acid (85 %, 20.0 g), sodium hydroxide (6.0 g), sodium silicate (8.0 g), and distilled water (30–40 mL) were used for the synthesis. The molar amounts of the reagents were calculated as follows: $n(\text{H}_3\text{PO}_4) = 0.1735 \text{ mol}$; $n(\text{NaOH}) = 0.1500 \text{ mol}$; $n(\text{Na}_2\text{SiO}_3) = 0.0655 \text{ mol}$. The overall Na/P molar ratio is thus 1.62, the Na/P ratio attributable to the alkali alone is 0.86, and the Si/P ratio is 0.38. The calculated oxide composition of the reaction mixture is given in Table 1.

Table 1

Calculated oxide composition of the reaction mixture

Component	Amount, g	n, mol	Mass fraction, %	Mole fraction, %
Na ₂ O	8.71	0.1406	34.90	48.0
P ₂ O ₅	12.31	0.0867	49.33	29.6
SiO ₂	3.94	0.0655	15.78	22.4
Total	24.96	—	100.00	100.0

Note: The calculation was performed for anhydrous Na₂SiO₃ (M = 122.06 g/mol).

The synthesis was carried out in the following sequence. In the first stage, 6.0 g of NaOH was dissolved in 20 mL of distilled water, and the solution was allowed to cool to room temperature. In the second stage, 20.0 g of orthophosphoric acid was stirred on a magnetic stirrer, and the NaOH solution was added dropwise; the temperature was maintained at 40–60 °C, and stirring was continued for 20–30 min. In the third stage, 8.0 g of sodium silicate was added gradually to the resulting neutralised solution; the temperature was raised to 70–80 °C and stirring was continued for 60 min. In the fourth stage, the resulting mass was concentrated by evaporation and dried at 120–150 °C for 2 h. In the fifth stage, the dried product was placed in a porcelain crucible and polycondensed in a muffle furnace at 300 °C for 1 h, after which it was cooled slowly inside the furnace [10].

The thermal properties of the product were studied on a Shimadzu DTG-60 thermal analyzer. The sample mass was 16.831 mg, the crucible material was corundum (Al₂O₃), the atmosphere was flowing argon at 100 mL/min, the heating rate was 10 °C/min, and the temperature range was 33–800 °C (the maximum temperature reached by the sample was 775.97 °C). The mass-loss steps were determined from the initial and final points of the TG curve, and the maximum rates of the individual processes from the minima of the DTG curve. The instrument software does not output a DTG curve, so it was computed from the

primary data file. In order to suppress the noise arising from the discreteness of the balance signal, the DTG curve was calculated as the derivative with respect to temperature of the mass values smoothed with a Gaussian filter ($\sigma = 7\text{ }^{\circ}\text{C}$).

Results and discussion

The original TG and DTA record of the sample, as output by the Shimadzu DTG-60 software, is shown in Fig. 1. The curve referred to the left-hand axis is the thermogravimetric trace in milligrams, and that referred to the right-hand axis is the differential thermal signal in microvolts; the step boundaries and the temperatures of the thermal effects marked on the plot were generated by the instrument itself.

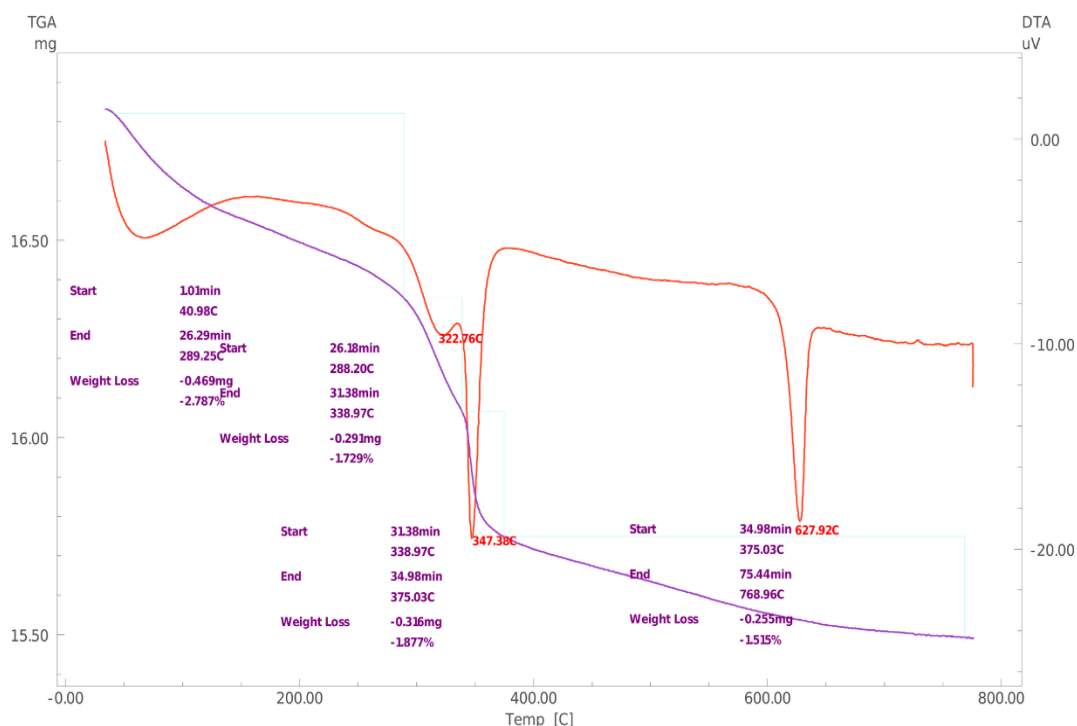


Fig. 1. Original TG and DTA record of the sample obtained on the Shimadzu DTG-60 thermal analyser (instrument output). Argon, 100 mL/min; heating rate 10 °C/min; sample mass 16.831 mg.

The primary record already reveals the general pattern of the process: a slow, continuous mass loss up to approximately 290 °C, a markedly accelerated loss between 290 and 375 °C accompanied by two endothermic effects, a further slow loss up to the end of the run, and finally a narrow, isolated endothermic effect near 628 °C that is not accompanied by any appreciable change in mass. Since the instrument software does not output a DTG curve, the differential mass-loss curve was computed from the primary data file and is presented together with the DTA curve in Fig. 2, which allows the rate maxima and the thermal effects to be compared directly on a common temperature scale.

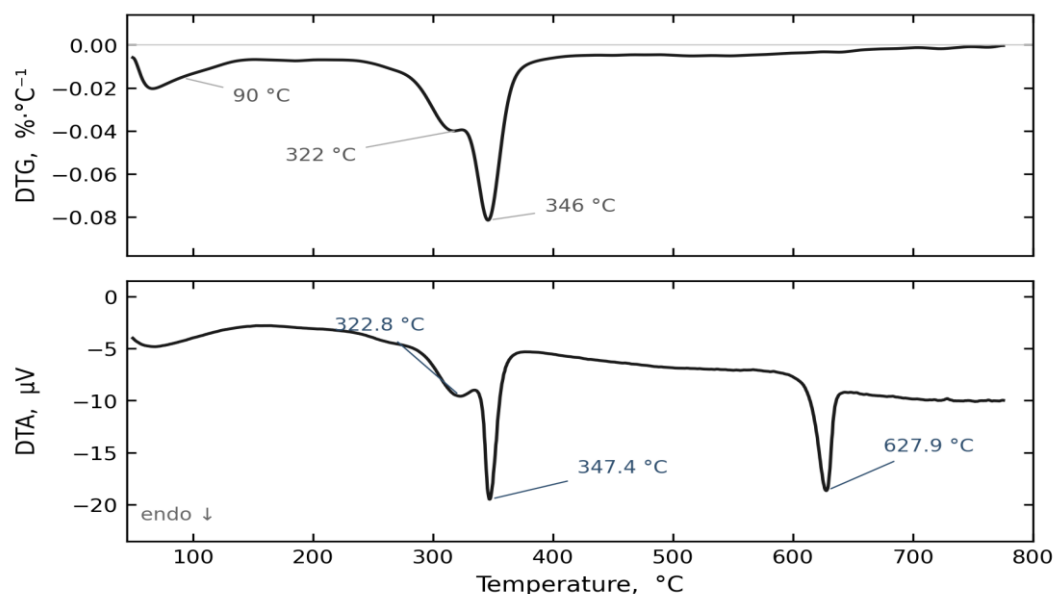


Fig. 2. DTG (top) and DTA (bottom) curves of the sodium polyphosphate–silicate composition, computed and replotted from the primary data of Fig. 1

Four clearly separated mass-loss steps can thus be distinguished on the TG curve up to 775 °C. Their quantitative characteristics are summarised in Table 2.

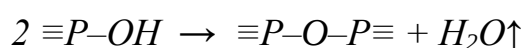
Table 2

Thermal decomposition steps of the sample

Step	Temperature range, °C	Δm , mg	Δm , %	DTG min, °C	DTA effect, °C
I	41–289	–0.469	–2.79	≈90	broad endotherm
II	289–339	–0.291	–1.73	322	322.8 (endo)
III	339–375	–0.316	–1.88	346	347.4 (endo)
IV	375–769	–0.255	–1.51	—	627.9 (endo)
Total	41–775	–1.341	–7.97	—	—

First step (41–289 °C, –2.79 %). The mass loss in this range is slow and continuous, and a flat minimum is observed on the DTG curve at approximately 90 °C. Such behaviour is characteristic of the removal of physically adsorbed and weakly bound water. The sample analysed had previously been heat-treated at 300 °C for 1 h; this water therefore accumulated through re-adsorption of atmospheric moisture after the product was removed from the muffle furnace. This confirms the high hygroscopicity typical of glassy sodium polyphosphates and indicates that the product must be stored in a sealed container.

Second and third steps (289–375 °C, –3.61 % in total). These two steps are closely interrelated and correspond to the release of water through the condensation of residual P–OH groups:



The maximum rate of this process is recorded on the DTG curve at 346 °C, with an additional shoulder-shaped minimum at 322 °C. The corresponding features on the DTA

curve are a weak endothermic effect at 322.8 °C and a sharp endothermic effect at 347.4 °C. Their endothermic character confirms the dehydration–condensation nature of the process.

The quantitative treatment of these steps yields a result of practical importance. The complete condensation of NaH_2PO_4 to NaPO_3 corresponds to a theoretical mass loss of 15.02 %. The mass loss of 3.61 % observed experimentally over 289–375 °C amounts to 2.00 mmol H_2O per gram of product. This shows that the heat treatment at 300 °C for 1 h achieved only about 76 % of the theoretically complete polycondensation, leaving on average 0.20 mol of uncondensed protons per phosphorus atom. The selected polycondensation regime is therefore insufficient; since the DTG maximum lies at 346 °C, it is recommended that the polycondensation temperature be raised to 350–400 °C or that the holding time be extended.

Fourth step (375–769 °C, –1.51 %). In the high-temperature region, the mass decreases very slowly and monotonically, and no distinct maximum is observed on the DTG curve. This is attributed to the gradual removal of the residual, strongly bound water and to the redistribution of the polyphosphate chains, that is, to an increase in the average chain length.

Endothermic effect at 627.9 °C. The most notable feature of the DTA curve is the narrow, sharp endothermic effect at 627.9 °C. This effect proceeds with practically no change in mass: the mass loss between 600 and 650 °C amounts to only 0.18 %. The invariance of the mass shows that the effect is not a decomposition but a phase transition, namely melting. The observed temperature almost exactly coincides with the melting point of sodium metaphosphate (NaPO_3), reported in the literature as 628 °C. This reliably confirms that chain sodium polyphosphate has been formed as the principal phase in the synthesised product.

It is noteworthy that the melting temperature practically does not deviate from the value for pure sodium metaphosphate. This suggests that the silicon component may be segregated as a separate silicate phase rather than being appreciably incorporated into the polyphosphate chains. X-ray phase analysis and IR spectroscopy are required for a definitive confirmation of this hypothesis.

Overall, the residual mass of the sample at 775 °C amounts to 92.03 % of the initial mass. This indicates a high thermal stability of the product in an inert atmosphere and, in particular, the absence of any volatilisation of phosphorus(V) oxide.

Conclusions

1. A sodium polyphosphate–silicate composition was synthesised by a one-pot, low-temperature route from orthophosphoric acid, sodium hydroxide and sodium silicate; the calculated molar ratios of the reaction mixture are $\text{Na/P} = 1.62$ and $\text{Si/P} = 0.38$.

2. The thermal decomposition of the product proceeds in four steps, with the total mass loss up to 775 °C being 7.97 %.

3. The mass loss of 3.61 % over 289–375 °C corresponds to the polycondensation of residual P–OH groups and shows that heat treatment at 300 °C for 1 h provides only about 76 % of the theoretically complete condensation. In order to complete the process, raising the polycondensation temperature to 350–400 °C is recommended.

4. The sharp endothermic effect observed at 627.9 °C, which proceeds without any change in mass, coincides with the melting point of NaPO_3 (628 °C) and confirms the formation of a chain polyphosphate phase in the product.

5. The synthesised product is thermally stable in an argon atmosphere up to 775 °C, the residual mass being 92.03 %.

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