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Stability of Sodium Percarbonate in Quality Control of Detergent using Isothermal Microcalorimetry

Sodium percarbonate is manufactured in vast quantities around the world and is a major ingredient in washing powders and detergents. Unfortunately, sodium percarbonate is thermally unstable and undergoes thermal degradation according to the following scheme.

$$4\ NaCO_3(s) \cdot 1½\ H_2O\ (l) \leftrightarrow 4\ Na_2CO_3\ (s) + 3\ O_2(g) + 6\ H_2O\ (g)$$

This degradation pathway has two major consequences. The first is that the purity of the sodium percarbonate is compromised with water and sodium carbonate being produced. Water in the powder can alter the reology making it difficult to transport and pump around a plant. A second consequence is that the reaction is exothermic and produces oxygen. Under certain storage conditions, it is possible for the material to accumulate heat and self-heat. Self-heating in the presence of oxygen is not desirable to the safe operation of a manufacturing plant. Thus, the heat formed during degradation is a problem for both the raw material manufacturer as well as the detergent producer since the safety on storage of bulk quantities of this product will be significantly reduced.

In order to assess the rate of degradation of sodium percarbonate, isothermal microcalorimetry has proved to be a rapid, simple and reliable tool. Several laboratories around the world have developed methods using this technique that give a qualitative assessment of sodium percarbonate reactivity. Below, the method utilised by Procter and Gamble is described.

Experimental

A 2277 Thermal Activity Monitor is set to 40°C. Sodium percarbonate (2 g) is loaded into a 3 ml disposable crimp seal ampoule under ambient conditions. The crimp is applied and the cap adjusted to fit into the calorimeter. The sample is lowered into the calorimeter with an empty ampoule serving as a reference. After 30 minutes the calorimeter will be in thermal equilibrium. Data points are collected every 60 seconds for a period of 48 hours.

Results and Discussion

The specific heat flow after 16 and 48 hours is assessed. If the specific heat flow is less than 10 µW/g at both these time points, the sodium percarbonate under test is considered to be stable enough for use.

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Fields of application
Materials Science
Quality control

Instrument configuration
2277 Thermal Activity Monitor
5510 Pre-thermostat
2277-201 4ml Ampoule Microcalorimetric Unit
2277-131 Digitam 4.1 for Windows
2277-303 3ml Disposable Glass Ampoules

Typical microcalorimetric responses for sodium percarbonate stability tests are shown in Figure 1 and the results tabulated in Table 1.

Conclusions

Isothermal microcalorimetry has proved to be an excellent tool in the quality control of sodium percarbonate stability in the detergent industry. The test is simple, unambiguous, non-labour intensive, rapid and robust. The method is increasingly becoming the industry standard for sodium percarbonate stability.

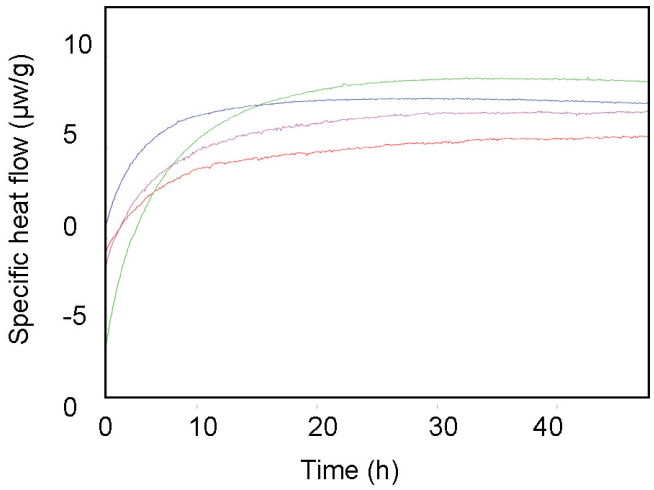

Figure 1. *Typical microcalorimetric responses for sodium percarbonate stability tests at 40°C.*

Sample	Specific Heat Flow at 16 Hours (µW/g)	Specific Heat Flow at 48 Hours (µW/g)	Pass/Fail
1	4.4	5.6	Pass
2	7.5	8.6	Pass
3	7.3	7.4	Pass
4	6.0	6.9	Pass

Table 1. *Heat flow values calculated at 16 and 48 hours at 40°C for a sample of sodium percarbonate. Data calculated from Figure 1.*