# Synthesis of energy saving integrated flowsheet for sodium hypophosphite production

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#### 1. Introduction

The process of sodium hypophosphite (SH) production (Fig .1) consists of several basic stages, as follows:

- preparation of initial components for SH synthesis and their supply to main reactor;
- synthesis of sodium hypophosphite;
- separation of sodium hypophosphite as end product;
- heat energy supply and cooling (Lisitsina, 1994, EU ECOPHOS Project, 2008).

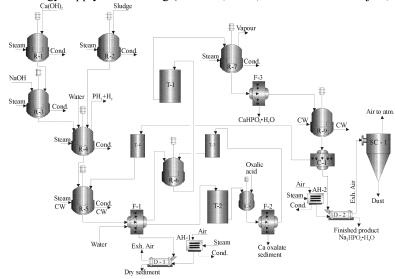


Figure 1. Principal flowsheet for sodium hypophosphite production. R1 - reactor for Ca(OH)2 preparation; R2 - reactor for phosphoric sludge preparation; R3 - NaOH-Ca(OH)2 preparation; R4 - main reactor; R5 - following reactor; R6 - neutralization; R7 - evaporator; R8 - sedimentation by oxalic acid; R9 - crystallization; F1-3 - filter; T1-4 - tank; C1 - centrifuge; SC1 - cyclone; D1,2 - dryer; AH1,2 - air heater.

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The aim of preparation stage is to make NaOH-Ca(OH)<sub>2</sub> suspension and to prepare phosphorous-containing sludge. Synthesis unit consists of main reactor and following reactor. After filtration the obtained solution of SH is corrected in neutralizer. Separation stage is the most complicated stage from viewpoint of equipment involved. The objective of separation stage process is to obtain the sodium hypophosphite of commercial grade from sodium hypophosphate which is coming after synthesis stage. The process of sodium hypophosphite production is complicated chemical-technological system which includes chemical reaction, separation system of obtained products, utility system. But heat recovery system is absent in this process. It is necessary to estimate possibilities of heat integration with the use of process integration methods. Principal flowsheet of sodium hypophosphite production is presented on Figure 1.

## 2. Heat integration of sodium hypophosphite production

#### 2.1 Energy saving potential estimation

There is no recovery of heat energy between technological streams in existing flowsheet. Evidently it is necessary to build new heat exchanger network on the base of pinch design principles (Linnhoff B. and Ahmad S., 1990, Linnhoff B. and Hindsmarsh E., 1983). For grass root design it is required to define  $\Delta T_{min}$  between process streams and targets of cold and hot utilities. For this we carried out the data extraction of existing process and found all needed stream data.

Utility targets can be obtained from composite curves. But at first we should build cost curves of sodium hypophosphite production heat system to define optimal value of  $\Delta T_{min}$ . To draw the cost curves it is necessary to know costs of hot utility, cold utility, cost of 1  $m^2$  of heat exchange surface, cost of heat exchanger installation and cost law. Cost of heat exchange surface depends on such factors: stream aggressiveness, impurities, pressure drop and other. Analysis of process flowsheet has shown that there are 2 main reactors, 4 following reactors and 2 evaporation units. It allows to consider this process like continuous one.

In existing process hot streams duty is 7605 kW and cold stream duty is 9782 kW.

For economically optimal integration the main economic indicators which influence the project reduced cost should to be defined. Cost of hot utilities is accepted at 200 USD per 1 kW year, it correspond to 170 USD for 1000 m³ of natural gas and for 8000 working hours per year. Cost of cold utilities is accepted as 0.1 from cost of hot utilities, i.e. 20 USD per 1 kW year. Specific price of heat-transfer area is taken equal to 1000 USD/m². Installation costs with remanifolding of 1 heat exchanger is 10 000 USD. For accomplishment of the retrofit project the enterprise takes loan for 5 years with interest rate 10%.

Calculation of project cost for sodium hypophosphite production allows to define minimal temperature difference for heat recovery system  $\Delta T_{min}=10^{\circ}$ . Modern tendencies of energy carriers market show price rising for some next decades (Klemes, Bulatov, 2007). Therefore dependence for optimal  $\Delta T_{min}$  of heat recovery system from energy prices should be considered.

Analysis of dependence of optimal  $\Delta T_{min}$  from energy price was made for energy prices from 100 USD per 1 kW year to 400 USD per 1 kW year. Cold utility price was varied from 10 to 40 USD per kW year. Figure 2 shows that there are no big changes of  $\Delta T_{min}$  localization under energy price variation. More detailed analysis shows that optimal  $\Delta T_{min}$  is slightly modified within utility prices changing range. Optimal  $\Delta T_{min}$  is changed from 20°C to 4°C within utility price change from 100 USD per kW year to 400 USD per kW year.

Dependence of recovery system project cost from minimal temperature difference of heat carriers in heat exchangers shows small changes on the interval  $\Delta T_{min} \in 5...20^{\circ} C$  for all investigated range of energy costs. Taking all above into account, we can conclude that heat recovery system of sodium hypophosphite production designed for  $\Delta T_{min} \in 5...20^{\circ} C$ , for a example  $\Delta T_{min} = 10^{\circ} C$ , will work in optimal mode.

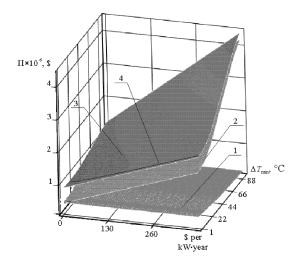


Figure. 2. Capitalized values vs.  $\Delta T_{min}$  and specific energy cost: 1– capitalized value for equipment; 2 – capitalized value for energy; 3 – common capitalized value; 4 – extreme value of annual costs.  $\Delta T_{min\ opt}$ =10 °C for all range of energy costs.

Composite curves plotted for  $\Delta T_{min}=10^{\circ}C$  shows heat power which is needed to be supplied for the integrated process functioning,  $Q_{Hmin}=4414.6$  kW (see Fig. 3). This value is on 5377.2 kW less than heat power for process without recuperation system. Thus process integration reduces heat power consumption on 55%, or more than two times. The requirement for cold utilities will be  $Q_{Cmin}=2237.9$  kW. It is on 70% less than for process without heat integration. Pinch temperature for hot streams is  $T_{Hpin}=108^{\circ}C$  and for cold streams is  $T_{Cpin}=98^{\circ}C$ . Estimated heat-transfer area for heat recovery system is equal S=3050 m² and number of heat exchangers is 16. Therefore pinch project of heat exchanger network will be created for  $\Delta T_{min}=10^{\circ}C$ . If to set pinch temperatures of hot and cold streams ( $T_{Hpin}=108^{\circ}C$ ,  $T_{Cpin}=98^{\circ}C$ ) on technological streams population (see Fig. 4), we can see that all technological streams are situated below the pinch point except streams No.13 and No.14.

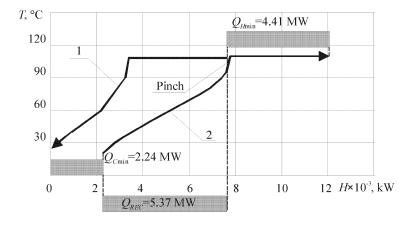


Figure. 3. Composite curves for integrated process at the  $\Delta Tmin = 10$  °C: I- hot composite curve; 2 -cold composite curve. QHmin - hot utility; QCmin - cold utility; QREC - recuperation duty

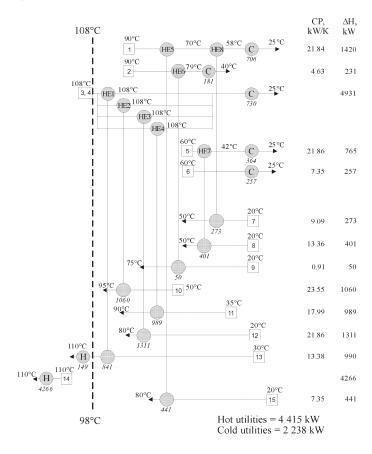


Figure. 4. Grid diagram of integrated process for hypophosphite production with the  $\Delta T_{min}$  =10 °C

If to consider pinch temperatures for  $\Delta T_{min}$ =4°C they are equal to  $T_{Hpin}$ =108°C,  $T_{Cpin}$ =104°C. If these temperatures are set on grid diagram, structure of process streams population will be invariant concerning pinch point localization, i.e. topology of heat recovery system will be constant for selected variation of  $\Delta T_{min}$ . It shows that in this case pinch project can also be created as for  $\Delta T_{min}$ =10°C.

#### 2.2 Pinch project for sodium hypophosphite production process

During pinch design only project below pinch point have to be accomplished because above the pinch point there are only 2 cold streams. On these 2 streams hot utilities should to be placed (Linnhoff and Flower, 1978, Rev and Fonyo, 1982). After the accomplishing pinch designing for stream system below the pinch point, grid diagram of heat exchanger network for sodium hypophosphite production process looks like it is shown on Figure 4.

In obtained project pinch crossing is absent and defined earlier energy targets are achieved. Using grid diagram lets built principle process flowsheet of sodium hypophosphite production. Making process flowsheet we will use existing heat exchange surfaces. Existing air heaters AH-1 and AH-2 can be used in the offered project on the same position HE-3 and HE-5. Stream 7 can be heated via steam jacket of the reactor R-1 by stream1 after heat exchanger HE-5. Stream 10 (phosphoric sludge decomposition) is heated by part of stream 3 in steam jacket of reactor R4. Principle flowsheet of sodium hypophosphite production with 4 additional heat exchangers (see Fig. 5) was obtained after accomplishing pinch design with mentioned remarks.

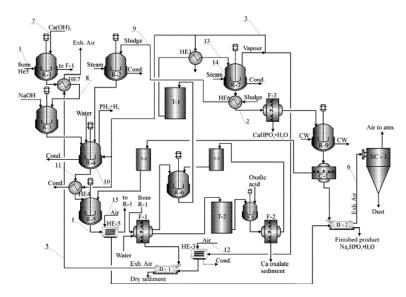


Figure. 5. Principal flowsheet for integrated sodium hypophosphite production. R1 - reactor for  $Ca(OH)_2$  preparation; R2 - reactor for phosphoric sludge preparation; R3 - preparation  $NaOH-Ca(OH)_2$  preparation; R4 - main reactor; R5 - following reactor; R6 - neutralization; R7 - evaporator; R8 - sedimentation by oxalic acid; R9 - crystallization; F1-3 - filter; F1-4 - tank; F1-4 - tank; F1-4 - centrifuge; F1-4 - cyclone; F1-4 - dryer; F1-4 - heat exchangers.

Comparison of energy consumption of sodium hypophosphite production with and without recovery system is presented in table 1.

Table 1. The comparison of energy consumption for project with heat integration and without heat integration.

Project	Hot utilities, kW	Cold utilities, kW	Recuperation, kW	Cost of energy during the year, USD
Existing process	9791.8	7605.1	0	2 108 000
Integrated process	4414.6	2237.9	5367.2	927 000

#### 3. Conclusion

The significant potential for energy saving in sodium hypophosphite production using pinch technology is shown. Economy due to application of energy recovery system is 1 200 000 USD. Installation of new recuperative heat exchangers will cost about 1 400 000 USD and payback period is 14 months.

### 4. Acknowledgments

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