

JÓZEF KUROPKA*, MIECZYŚLAW A. GOSTOMCZYK*

ABSORPTION OF NITROGEN OXIDES ALKALINE ABSORBENTS

The utility of some alkaline solutions, when applied to the treatment of flue gases (removal of nitrogen oxides) from large and small industrial plants, is estimated. The effect of some major parameters (gas velocity, initial concentration of nitrogen oxides in the flue gas, concentration and type of the spraying solution, spraying density, type and depth of the packing, pressure loss) on the kinetics of absorption is considered. The contribution of oxidizing (or reducing) additives in the alkaline solutions on the absorption efficiency is determined. Correlations (in the form of the function $\eta = 1 - \exp[-f(\text{process variables})]$) generalizing the results obtained have been derived.

1. INTRODUCTION

Successful applications of alkaline absorption for the needs of environmental pollution control depend on the physicochemical properties of nitrogen oxide and nitrogen dioxide. ATROSHCHENKO [1] has found that absorption in alkaline solutions runs very fast when a mixture of nitrogen oxides at a voluminal ratio of nitrogen dioxide to nitrogen oxide equal to (or higher than) unity is involved. Specialized literature contains many references to the absorbing solutions that are in use now. Here are some major examples: sodium hydroxide [2]–[7], ammonium hydroxide [4], [8], potassium hydroxide [2], sodium carbonate (most frequently used) [9]–[11] or ammonium carbonate [12], [13], [14]. Nitrogen oxides carried by flue gases can also be absorbed in aqueous slurries of magnesium hydroxide [15] or calcium hydroxide [16].

To improve the efficiency of removal, absorption of nitrogen oxide was carried out in aqueous mixed solutions containing strong oxidants, e.g. sodium hypochlorite [17]–[19], sodium chlorite [20], [21], [22], calcium hypochlorite [23], [24],

* Institute of Environment Protection Engineering, Technical University of Wrocław, Wybrzeże Wyspiańskiego 27, 50-370 Wrocław, Poland.

potassium permanganate and potassium dichromate [25]–[27], iron, copper, nickel and cobalt salts [28], [29], or hydrogen peroxide [30]–[32]. When use is made of the reducing properties of alkaline solutions containing sulphides, sulphites or thiosulphites, nitrogen oxides can be removed in the form of free nitrogen. In terms of environmental pollution control, this is the most advantageous form of removal. No wonder that the need for more efficient removal of nitrogen oxides from industrial gases has directed the attention of many investigators to those processes. Thus, apart from absorbing solutions that contain sulphites alone [33]–[37], use is also made of some additives, viz. iron salts [38], copper, cobalt and nickel salts [39] or Fe-EDTA chelates [40]–[45].

2. EXPERIMENTAL APPARATUS AND METHODS

The experimental system and the experimental conditions have been described elsewhere [46]. To investigate alkaline absorption of nitrogen oxides the following solutions have been used [47]:

- a) NaOH, KOH, NH_4OH , Na_2CO_3 , K_2CO_3 , $(\text{NH}_4)_2\text{CO}_3$,
- b) NaOH with an oxidizing additive: NaOCl , H_2O_2 , KMnO_4 , $\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4$, $\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4 + \text{EDTA}$, $\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4 + \text{EDTA} + \text{CH}_3\text{COOH}$,
- c) NaOH with a reducing additive: Na_2SO_3 , $(\text{NH}_4)_2\text{SO}_3$, Na_2S , $(\text{NH}_4)_2\text{S}$.

The objectives of this experimental series can be itemized as follows:

1. To verify the potentiality for alkaline absorption (in aqueous solutions of hydroxides and carbonates; in alkaline solutions with oxidizing or reducing agents) in the treatment of flue gases from large industrial plants (manufacture of nitric acid; manufacture of sulphuric acid by mixed acid method) or from small emission sources (electroplating, etching, etc.).

2. To determine to effect of some major parameters (gas velocity; concentration of nitrogen oxides in the flue gas stream; concentration and type of the spraying solution; spraying density; type and depth of the column packing; pressure loss) on the kinetics of absorption.

3. To establish the contribution of oxidizing agents in the alkaline solutions to the kinetics of the absorption process.

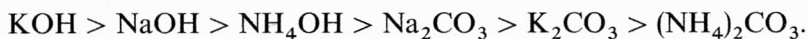
4. To determine the effect of reducing agents in the alkaline solutions on the removal efficiency.

The experiments were run in series which included a dozen or more measuring procedures. In each series the type and depth of the packing, the concentration of nitrogen oxides in the flue gas ($\text{NO}_2:\text{NO} = 1$), the concentration of the spraying solution, and spraying density were kept constant (gas velocity being a variable parameter). Curves were plotted from the results of three experiments for a given gas velocity.

3. RESULTS AND DISCUSSION

3.1. ABSORPTION OF NITROGEN OXIDES IN AQUEOUS SOLUTIONS OF HYDROXIDES AND CARBONATES

Comparing the results obtained with the investigated solutions of hydroxides and carbonates, we can arrange them as follows (according to the descending order of activity):



The contribution of the absorbent concentration to the kinetics of the absorption process varies from one alkaline solution to another. The results are plotted in figs. 1 and 2.

As shown by these data, the concentration of the solution has a substantial influence on the efficiency of absorption up to the value of 2 wt.% in the case of sodium hydroxide (fig. 1). In the case of sodium carbonate, the concentration effect is noticeable up to the value of 10 wt.% (fig. 2). Applications of concentrations higher than 2 wt.% and 10 wt.%, respectively, are only recommended when the effluent from the absorption process is to be utilized.

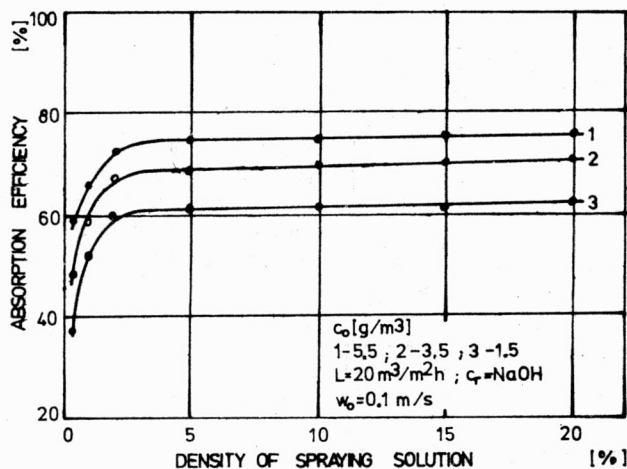


Fig. 1. Absorption efficiency versus sodium hydroxide solution concentration, at constant spraying density ($20\text{ m}^3/\text{m}^2\text{ h}$) and constant gas velocity (0.1 m/s) for various initial NO_2 concentrations

1 - $c_0 = 5.5\text{ g/m}^3$, 2 - $c_0 = 3.5\text{ g/m}^3$,
 3 - $c_0 = 1.5\text{ g/m}^3$

Absorption efficiency was also related to gas velocity, spraying density, as well as to the type and depth of the column packing (figs. 3 and 4). The plots are similar in shape for all the alkaline solutions under study. The contribution of initial NO_2 concentration in the flue gas stream is particularly distinct up to the value of 5.5 g/m^3 ($0.27\text{ vol.}\%$). Once this value has been exceeded, the relation between absorption efficiency and initial NO_2 concentration is of linear nature. When gas velocity increases (the remaining parameters being kept constant), the efficiency of the process decreases. The increase in spraying density brings about an increase in

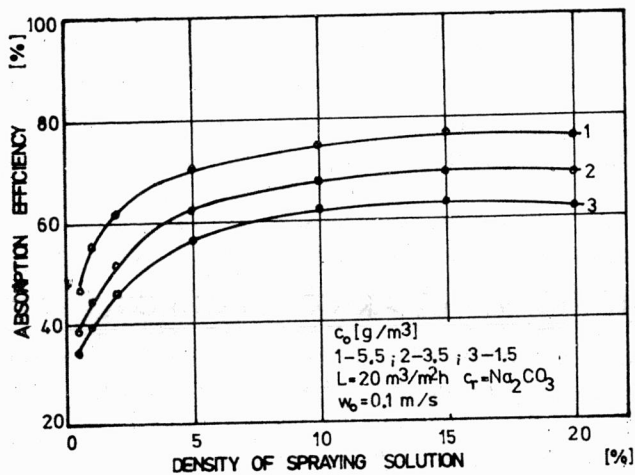


Fig. 2. Absorption efficiency versus sodium carbonate solution concentration, at constant spraying density ($20 \text{ m}^3/\text{m}^2 \text{ h}$) and constant gas velocity (0.1 m/s) for various initial NO_2 concentrations

1 - $c_0 = 5.5 \text{ g/m}^3$, 2 - $c_0 = 3.5 \text{ g/m}^3$,
3 - $c_0 = 1.5 \text{ g/m}^3$

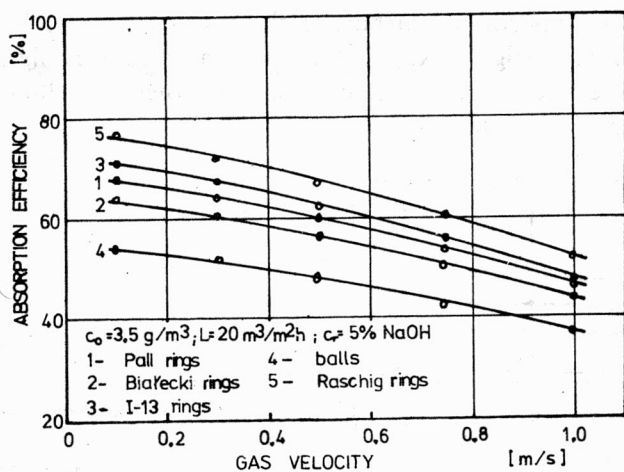


Fig. 3. Absorption efficiency versus packing type at constant initial NO_2 concentration ($c_0 = 3.5 \text{ g/m}^3$) for 5 wt.% sodium hydroxide solution and constant spraying density ($20 \text{ m}^3/\text{m}^2 \text{ h}$)

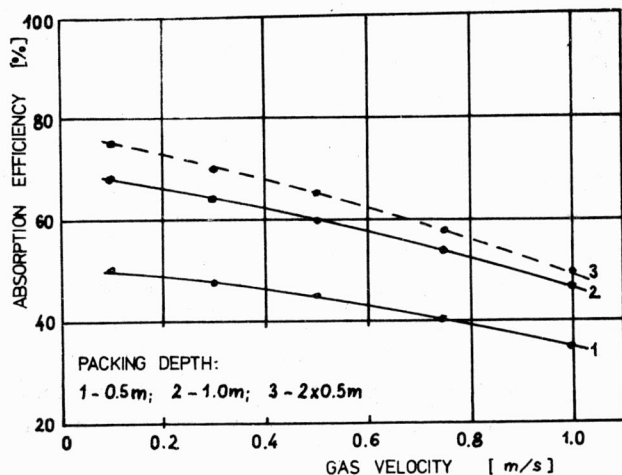


Fig. 4. Absorption efficiency versus gas velocity for various packing depth (Pall rings) at constant initial NO_2 concentration ($c_0 = 3.5 \text{ g/m}^3$), constant sodium hydroxide solution concentration (5 wt.%) and constant spraying density ($20 \text{ m}^3/\text{m}^2 \text{ h}$)

absorption efficiency (the optimum spraying conditions being achieved at a density of $20 \text{ m}^3/\text{m}^2 \text{ h}$). The application of various depths and types of packing has revealed that, as the time of contact increases, so does the efficiency of the absorption process. The application of inter-layer spraying (fig. 4) and the increase in the specific surface of the packing account for the improvement of the absorption effect by several percent. The only drawback of applying packings with increased specific surface areas is an increased pressure loss (fig. 5) which unfavourably affects the economics of the treatment system [48], [49].

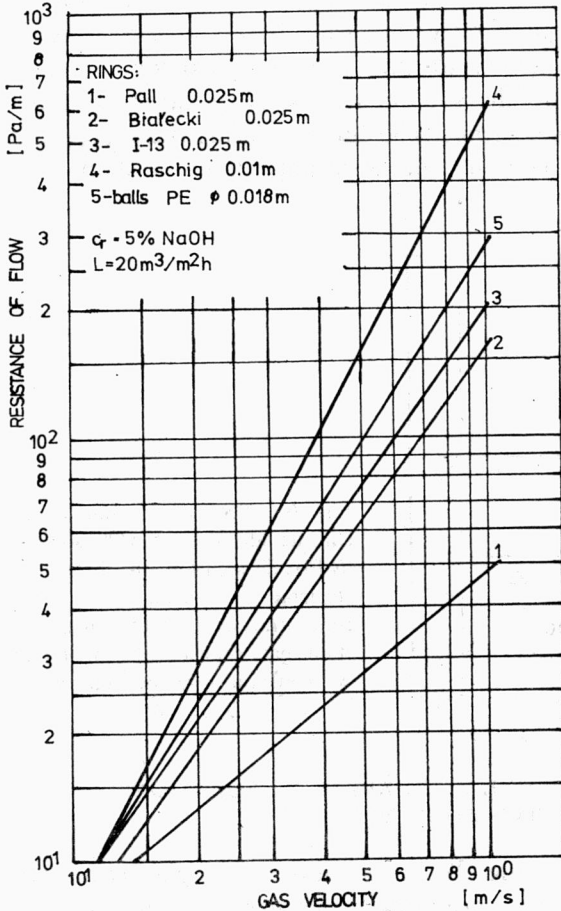


Fig. 5. Pressure loss versus gas velocity for various packing at constant spraying density ($20 \text{ m}^3/\text{m}^2 \text{ h}$)

3.2. ABSORPTION OF NITROGEN OXIDES IN ALKALINE SOLUTIONS WITH OXIDIZING ADDITIVES

This experimental series aimed at determining the contribution of oxidizing additives to the efficiency of the alkaline absorption method. The results are plotted

in figs. 6–9. The solutions used in the experiments showed a high absorbing capacity with respect to nitrogen oxides. These were sodium hypochlorite, hydrogen peroxide, potassium permanganate and iron complexes. The application of the 5 wt.% sodium hydroxide solution made it possible to compare the influence of selected oxidizing agents on the absorption of nitrogen oxides. Thus, for sodium hypochlorite this influence is noticeable up to the concentration of 2 wt.% (fig. 6). Hydrogen peroxide influences the absorption efficiency up to the concentration of 5 wt.% (fig. 7). The influence of potassium permanganate increases non-linearly with its concentration in the hydrogen oxide solution (fig. 8). Potassium permanganate in a potassium hydroxide solution improves the removal of nitrogen oxides by about 4–6% (fig. 9) under the same conditions as when added to the sodium hydroxide solution. Addition of ferroammonium sulphate to the sodium hydroxide solution accounts only for a slight increase in the removal efficiency. The curves relating the absorption efficiency to the major process parameter do not noticeably differ in shape.

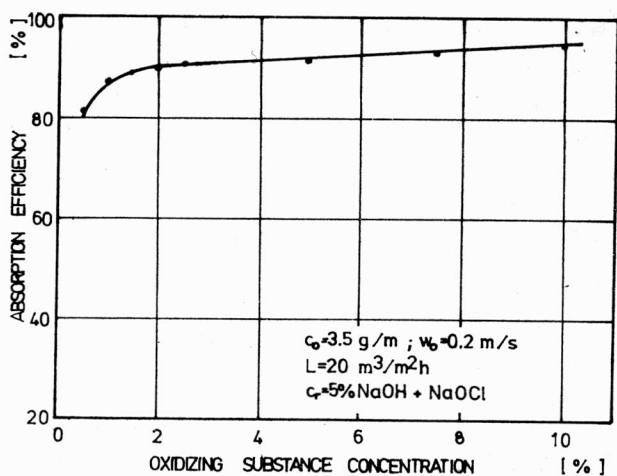


Fig. 6. Effect of sodium hypochlorite additive in 5 wt.% sodium hydroxide solution on absorption efficiency at constant initial NO_2 concentration ($c_0 = 3.5 \text{ g/m}^3$), constant gas velocity (0.2 m/s) and constant spraying density ($20 \text{ m}^3/\text{m}^2 \text{ h}$)

3.3. ABSORPTION OF NITROGEN OXIDES IN ALKALINE SOLUTIONS WITH REDUCING ADDITIVES

When use is made of sulphide or sulphite alkaline solutions, nitrogen oxides are removed in the form of free nitrogen. This experimental series was run primarily with a sodium hydroxide solution of 5 wt.% concentration. The reducing agents used in the experiments were sodium and ammonium sulphides, as well as sodium and ammonium sulphites. The experimental conditions were the same as those in the experimental series with oxidizing additives. The contribution of the reducing agent to the removal efficiency was noticeable for the entire range of the investigated variable parameters. The effect of sulphide on the absorption of nitrogen oxides

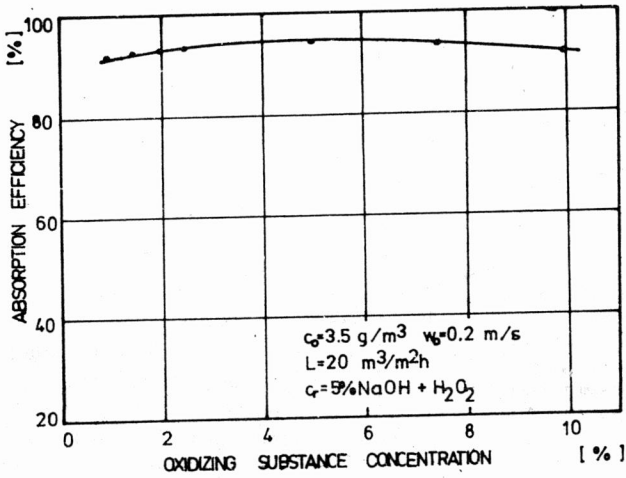


Fig. 7. Effect of hydrogen peroxide additive in 5 wt.% sodium hydroxide solution on absorption efficiency at constant initial NO_2 concentration ($c_0 = 3.5 \text{ g/m}^3$), constant gas velocity (0.2 m/s) and constant spraying density ($20 \text{ m}^3/\text{m}^2 \text{ h}$)

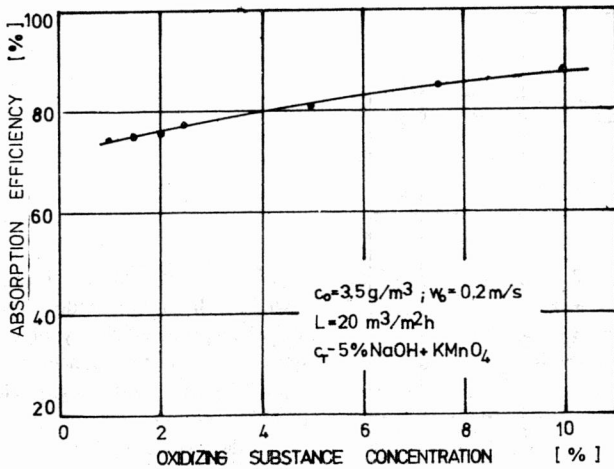


Fig. 8. Effect of potassium permanganate additive in 5 wt.% sodium hydroxide solution on absorption efficiency at constant initial NO_2 concentration ($c_0 = 3.5 \text{ g/m}^3$), constant gas velocity (0.2 m/s) and constant spraying density ($20 \text{ m}^3/\text{m}^2 \text{ h}$)

is plotted in fig. 10. As shown by these data, absorption efficiency increases rapidly with the increasing sulphide concentration. Once the value of 2 wt.% is exceeded, there is a slight linear increase in the process efficiency with the increase of sulphides concentration in the spraying solution. The difference in the plots between the two types of sulphides is insignificant (only removal efficiency is by about 5% higher when sodium sulphide is involved).

Figure 11 relates the removal efficiency to the concentration of sulphite in the sodium hydroxide solution. Thus, in the investigated concentration range (0.5–10 wt.%), the curves for sodium sulphite and ammonium sulphite display different

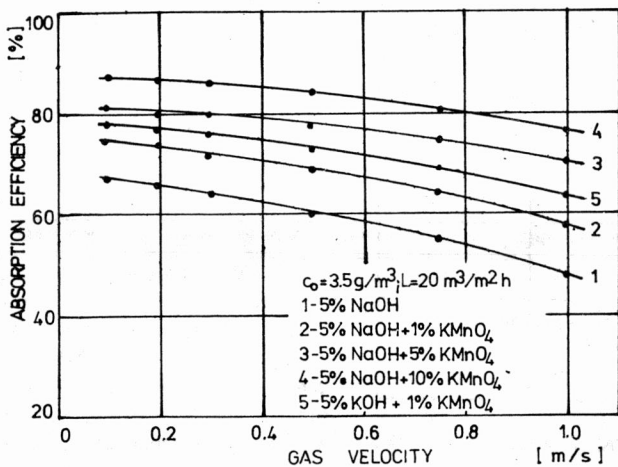


Fig. 9. Absorption efficiency versus gas velocity for absorbing solutions of various potassium permanganate content at constant initial NO_2 concentration (3.5 g/m^3) and constant spraying density ($20 \text{ m}^3/\text{m}^2 \text{ h}$)

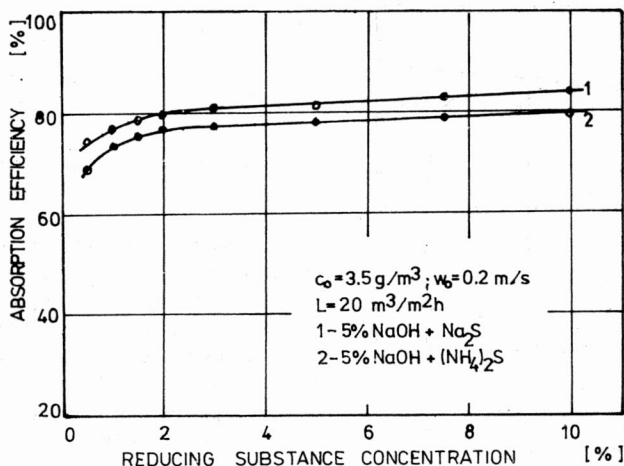


Fig. 10. Effect of sulphites in 5 wt.% sodium hydroxide solution on absorption efficiency at constant initial NO_2 concentration ($c_0 = 3.5 \text{ g/m}^3$), constant gas velocity (0.2 m/s) and constant spraying density ($20 \text{ m}^3/\text{m}^2 \text{ h}$)

shapes. The contribution of sodium sulphite increases noticeably up to the concentration value of 4 wt.%; that of ammonium sulphite rises rapidly up to the concentration value of 2 wt.%. When these values are exceeded, the contribution of the two reducing agents increases slightly in a linear manner with the increasing concentration of sulphites in the absorbent. The efficiencies of nitrogen oxide removal average by 8 to 10% higher for alkaline solutions of ammonium sulphite. In the investigated range of variable parameters, absorption efficiency increases with the increasing initial NO_2 concentration, decreases with the increasing gas velocity (at constant spraying density), and increases with the increasing spraying density (at constant gas velocity).

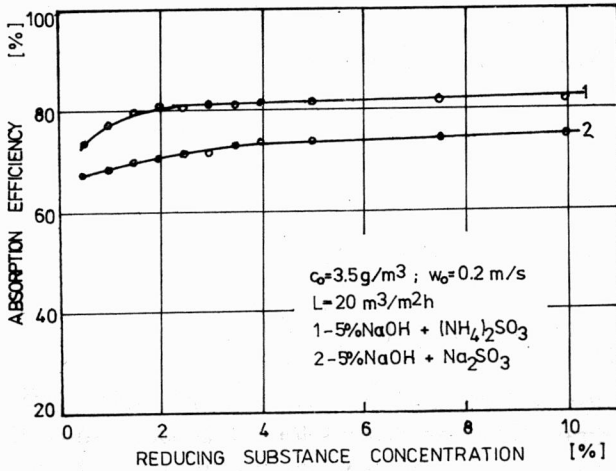


Fig. 11. Effect of sulphites in 5 wt.% sodium hydroxide solution on absorption efficiency at constant initial NO_2 concentration ($c_0 = 3.5 \text{ g/m}^3$), constant gas velocity (0.2 m/s) and constant spraying density ($20 \text{ m}^3/\text{m}^2 \text{ h}$)

4. CORRELATIONS

Of the various approaches to the mass transfer process [48]–[51], a quantitative description has been adopted. The description involves the following definition of absorption efficiency:

$$\eta = \left(1 - \frac{c_{av}}{c_0}\right) 100\%, \quad (1)$$

$$c_{av} = \frac{\sum_{n=1}^n c_n}{n} \quad (2)$$

where η denotes absorption efficiency, %; c_0 indicates NO_2 concentration in the gas after passage through the column, g/m^3 ; c_{av} is average NO_2 concentration in the gas after passage through the column, g/m^3 ; and n stands for the number of samples collected for analysis.

In terms of η it is easy to estimate the effect of the fundamental process parameters on the removal of pollutants from flue gases.

Absorption efficiency is a function of the following variables:

$$\eta = f(w_0, L, c_0, c_r) \quad (3)$$

where w_0 is gas velocity calculated for the void space; L denotes spraying density, $\text{m}^3/\text{m}^2 \text{ h}$; and c_r indicates the concentration of the absorbing solution, kg/m^3 or wt.%.

For the investigated range of variable parameters, the results were plotted as the following functions:

$$\eta = f(w_0), \quad \eta = f(L), \quad \eta = f(c_0), \quad \eta = f(c_r). \quad (4)$$

On the basis of these plots, the efficiency of mass transfer can be written as

$$\eta = 1 - \exp[-f(\text{process variables})]. \quad (5)$$

After transformation we obtain

$$\ln \frac{1}{1-\eta} = f(w_0, L, c_0, c_r). \quad (6)$$

For eq. (6) the following function has been proposed:

$$\ln \frac{1}{1-\eta} = B_0(w_0)^{B_1} (L)^{B_2} (c_0)^{B_3} (c_r)^{B_4}. \quad (7)$$

The coefficients B_i have been determined by the linear multiple regression method, using a computer program. The correlations for the generalization of the experimental results have been calculated for the absorbing solutions that might be of utility in removing nitrogen oxides from industrial gases.

For the investigated range of variable process parameters, the absorption of nitrogen oxides in sodium hydroxide solutions can be described by the following relation

$$\ln \frac{1}{1-\eta} = 0.164 (w_0)^{-0.220} (L)^{0.260} (c_0)^{0.350} (c_r)^{0.145} \quad (8)$$

for which the multiple correlation coefficient and the mean error amounts to $R = 0.95$ and $\bar{x} = 5.97\%$, respectively.

For the absorption of nitrogen oxides in sodium carbonate solutions, the relation becomes

$$\ln \frac{1}{1-\eta} = 0.141 (w_0)^{-0.218} (L)^{0.222} (c_0)^{0.352} (c_r)^{0.232}. \quad (9)$$

From the correlations (8) and (9) it follows that the concentration of nitrogen oxides in the flue gas stream, the concentration of the absorbing solution, and spraying density exert a favourable effect on the absorption efficiency. This is not true for the increase in gas velocity which exerts a limiting effect.

The absorption of nitrogen oxides in alkaline hypochlorite solutions is described by

$$\ln \frac{1}{1-\eta} = 0.682 (w_0)^{-0.152} (L)^{0.229} (c_0)^{0.068} (c_r)^{0.033} \quad (10)$$

for which the multiple correlation coefficient and mean error are $R = 0.88$ and $\bar{x} = 4.91\%$, respectively.

The contribution of the reducing additive in the spraying solution to the removal efficiency has been illustrated on the example of nitrogen oxides absorption in an alkaline solution of sodium sulphite.

For the investigated range of variable parameters we have

$$\ln \frac{1}{1-\eta} = 0.229 (w_0)^{-0.054} (L)^{0.484} (c_0)^{0.039} (c_r)^{0.044} \quad (11)$$

with $R = 0.8$ and $\bar{x} = 4.91\%$.

The relations (10) and (11) show that spraying density contributes significantly to the efficiency of removal in both instances. This contribution is more pronounced in the presence of reducing additives. Initial concentration of nitrogen oxides has a similar effect on the removal efficiency in the presence of oxidizing or reducing agents. The contribution of gas velocity is more distinct when oxidizing substances are added.

5. SUMMARY

The absorption of nitrogen oxides in alkaline solutions can be carried out:

a) in sodium hydroxide or sodium carbonate solutions for the removal of nitrogen oxides from the flue gas stream emitted during manufacture of sulphuric acid by the mixed acid method;

b) in the solution of sodium hypochlorite for the treatment of flue gases from small industrial plants (e.g., from electroplating);

c) in sodium sulphide solution or ammonium sulphite solution for the treatment of flue gases from small industrial plants (e.g., from organic synthesis).

The investigations made it possible to develop nitrogen oxides removal technologies for both large and small industrial plants [47], [52]. The technologies proposed provide removal of nitrogen oxides via a simple system involving acceptable investment and operating costs.

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ABSORPCJA TLENKÓW AZOTU

BADANIA ABSORPCJI TLENKÓW AZOTU W ROZTWORACH ALKALICZNYCH

Stwierdzono przydatność wybranych roztworów alkalicznych do unieszkodliwienia tlenków azotu z gazów odlotowych, zarówno z dużych, jak i z małych zakładów przemysłowych. Określono wpływ zmian podstawowych parametrów, takich jak prędkość gazu, stężenie tlenków azotu w gazie oczyszczanym, stężenie i rodzaj roztworu oraz gęstość zraszania, rodzaj i wysokość wypełnienia oraz opory przepływu gazu, na kinetykę procesu absorpcji tlenków azotu. Określono również wpływ substancji utleniających (lub redukujących) w roztworach alkalicznych na sprawność oczyszczania gazów z tlenków azotu. Opracowano korelacje uogólniające wyniki badań absorpcji alkalicznej w postaci funkcji $\eta = 1 - \exp[-f(\text{zmienna procesu})]$.

АБСОРБЦИЯ ОКИСЕЙ АЗОТА ИССЛЕДОВАНИЯ АБСОРБЦИИ ОКИСЕЙ АЗОТА В ЩЕЛОЧНЫХ РАСТВОРАХ

Была установлена пригодность избранных щелочных растворов для обезвреживания окисей азота из отходящих газов, происходящих как из крупных, так и из малых промышленных предприятий. Определено влияние изменений основных параметров как: скорость течения газа, концентрация окисей азота в очищаемом газе, концентрация и род раствора, а также плотность орошения, род и высота заполнения, а также сопротивление течения газа, на кинетику процесса абсорбции окисей азота. Определено также влияние окисляющих (или редуцирующих) веществ в щелочных растворах на эффективность удаления окисей азота из газов. Разработаны зависимости, обобщающие результаты исследований щелочной абсорбции в виде функции $\eta = 1 - \exp[-f(\text{переменные процесса})]$.