

Magnetic Field Effects on Redox Potential of Reduction and Oxidation Agents

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Redox potentials of two reducing (sodium dithionite and glucose) and two oxidizing (hydrogen peroxide and sodium hypochlorite) agents were monitored at various concentrations and at different temperatures for 30–75 minutes after the exposure of their water solutions (glucose and hypochlorite solutions once; sodium dithionite and hydrogen peroxide solutions one, two and/or three-times) to the static magnetic field of flux density of 0.9 V s m^{-2} . The aim of the investigation was to suggest improvements, *i.e.*, intensification and stability, of the reduction-oxidation ability of selected agents applicable in textile fibre processing, primarily bleaching and vat dyeing. Results of the experiments show that magnetic treatment (of solutions) raises both the reducing ability of glucose and the oxidation ability of hydrogen peroxide and sodium hypochlorite, promising some technological and economical benefits for the textile industry as well as for other fields of chemistry.

INTRODUCTION

Environmental problems of the textile industry are mainly caused by discharges of wastewaters containing a variety of chemicals, dyes, acids, sodas and salts, as well as auxiliary products present in fibre processing, depending on the nature of the raw material and product. In general, a number of wet processes are involved, with high requirements for resource inputs, generating several types of wastewater, which are used in large amounts required for washing operations. Therefore, the target of many researchers has been to standardize the chemical processes used in the textile processing industry, and to develop low cost ones that would simultaneously improve or even upgrade the end-products.

Oxidation and reduction reactions are used to synthesize or chemically modify textiles, dyes and a multitude of other important processing compounds by changing the number of electrons assigned to atoms undergoing the reaction.

Oxidation, as the combination of a substance with oxygen, is a ubiquitous chemical reaction in the dye chemistry. Conversion of methyl groups into carboxylic acids or into styryl compounds is effected by various oxidants such as sodium hypochlorite, potassium permanganate, and sodium dichromate. Catalyzed oxidation of naphthalene by air leads to phthalic anhydride. Other important reactions include oxidation of leuco compounds to dyes¹ and the formation of complex polycyclic substances from simpler molecules.² Bleaching of natural fibre forming

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polymers (*e.g.*, cellulose and wool) is another process undertaken to obtain lighter coloured fibres and more commercially attractive products by removal of coloured components or to convert them to colourless substances.³ Conjugated double bonds responsible for absorption of light in the visible wavelength range can be eliminated either by reduction or oxidation. The lignin component in cellulosic fibres is the main colour determining characteristic. Chemical methods for bleaching of lignocellulosic fibres have been developed using different oxidation agents. Because of environmental concerns (formation of highly toxic chlorinated organic by-products, AOX), the use of chlorine dioxide based bleaching stages has been eliminated in most industrial bleaching procedures. Interest in totally chlorine-free bleaching processes has led to the development of peroxide based bleaching as a universal and cheap bleaching agent. However, its disadvantage, *i.e.*, achieving the optimum degree of hydrogen peroxide stability in order to exploit the full potential of the bleaching activity, has proposed systems applying transition metal ions and chelated metal activation. Further investigation of chemical aspects of textile fibres bleaching is needed, especially in low temperature wet processing without affecting the physical-mechanical properties of the bleached fibre products.

Similarly, reduction refers to a decrease in the amount of oxygen in a substance or its complete removal. In the coloration of textile fibres, vat (including indigo) and sulphur dyes, accounting for about 31 % of the world dyestuff consumption market,¹ have to be reduced (vatted) before the dyeing in order to be converted into the water-soluble *leuco* enolate form (Figure 1). To assure complete reduction, a proper reducing agent with a sufficiently low reduction potential should be chosen.⁴ Control of the reduction and oxidation processes can be carried out by measuring the oxidation-reduction potential (ORP), which represents the system potential of a solution. It is measured with the aid of an inert electrode that transmits potential. The electrode is usually platinum wire *vs* Ag/AgCl reference electrode with KCl electrolyte. The general form of the Nernst equation for ORP is:

$$\text{ORP} = E = E^\circ - \frac{RT}{nF} \ln \frac{[\text{reduced species}]}{[\text{oxidized species}]} \quad (1)$$

where E° is the standard redox potential ($\text{AgCl(s)} + e^- \rightarrow \text{Ag(s)} + \text{Cl}^-(\text{aq}); +0.222 \text{ V at } 25^\circ\text{C}$); R , T and F are, respectively, the gas constant, the absolute temperature and the Faraday constant and n is the number of electrons transferred.

The charge balance always sums to neutrality, but it is seen from the Nernst equation that as the concentrations of species able to donate electrons (reduced species) increase, the ORP decreases, and *vice versa*. Thus as oxygen or other oxidants are added to a solution, the

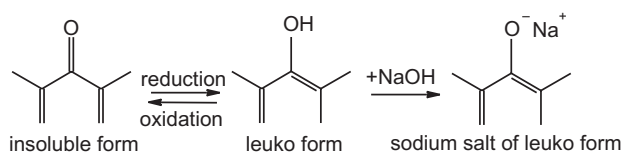


Figure 1. Redox reaction of conversion of dye insoluble to soluble form.

ORP rises or if the reducers are added, the ORP negatively decreases.⁵ Sodium dithionite is commonly used as reducing agent in vat dyeing, because it provides complete and rapid vatting and enables dyeing with highly set requirements. The problem with sodium dithionite is that the final products of oxidation (sodium sulphate, sulphite ions and thiosulphate ions) can damage the equipment because of their corrosiveness and when released detrimentally affect the environment due to their toxicity. Dyeing (absorption into the fibres) is followed by removal of the excess of reducing agents and sodium salts and by conversion of the dye back into its original water-insoluble pigment using oxidizing reagents, commonly hydrogen peroxide.^{6,7}

Environmental and economic considerations are strong motivations for developing alternative methods that would intensify the redox processes and reduce the consumption of chemicals. Accordingly, some attempts have been made in the textile industry with physical treatments, such as direct current, ultrasound and electromagnetic treatment.⁸ One of the physical treatments is the magnetic water treatment, which has been beneficially used for scale control in industrial water processing for the last two decades. Different types of magnetic devices can be found on the world market for this purpose. Some of them consist of differently arranged permanent magnets, others of different electromagnetic coils. The advantages of such devices are: low investment and easy installation, low energy consumption and ecological safety.

The practice of magnetic field applications in industry goes ahead of the theory of the phenomenon; namely, the mechanism of the magnetic-field influence on water systems is far away from the well-known magnetic separations, since applied magnetic fields are relatively weak (magnetic field densities are from 0.05 up to 1 V s m^{-2}) and treated compounds are mainly non-magnetic. In recent years, scientists have paid much attention to this field for both theoretical and practical purposes. Publications addressing this issue are increasing steadily every year, reporting that the magnetic field can influence a) the crystallization process in solutions^{9–11} as well as b) the electrokinetic potential of colloidal particles in electrolyte solutions.^{12,13} Two phenomena have been observed. First, when a water solution or dispersion flows through the magnetic field, the Lorentz force appears,

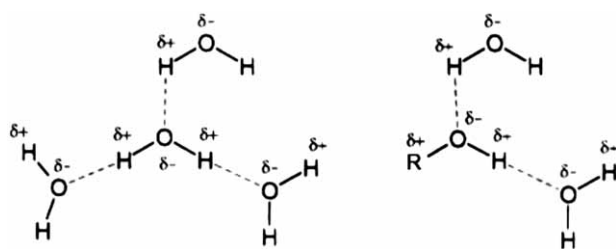


Figure 2. Hydrogen bonds in water and water solution of a substance with $-\text{OH}$ group.

acting on ions and dispersed particles, which are electrically charged. Hypothetically, this may result in changes in ions distribution at dispersed particles' surfaces, influencing the crystallization processes and colloidal stability.¹³⁻¹⁶ Second, a modification of physicochemical properties of water and water solutions (such as surface tension, dielectric constant and pH) occurs even during their static exposure to a static magnetic field. Joshi and Kamat¹⁷ reported the following changes of triple-distilled water caused by the exposure to a weak magnetic field (with field density from 0.19 to 0.57 V s m^{-2}): increase of pH up to 0.6, reduction of surface tension up to 16 mN m^{-1} , and an increase of the relative dielectric constant by 1.5. Although these changes are small and very sensitive to experimental conditions, they provided an important orientation for further research.

The results are assigned to distortion of the hydrogen bond (Figure 2). The hypothesis is that the magnetic field alters the magnetic spin of hydrogen protons in water by magnetic resonance. When two neighbouring protons in the net of water molecules transfer from anti-parallel into parallel pairs,¹⁸ some repulse appears between them and the H-bond gets distorted and weaker, affecting hydration and protonation of ions; if H-bonds get weaker by magnetic treatment, the hydration of ions is expected to be lower. Furthermore, some precise laboratory observations of magnetic modifications in crystallization,^{9,19} coagulation¹² and ion exchange²⁰ could be also explained by the changes in the hydration of ions and solid surfaces in water solutions.

Considering the mechanism, two types of magnetic treatments should be distinguished:

- static treatment, where water solution or dispersion is statically exposed to a static magnetic field,⁹⁻¹¹ and
- dynamic treatment, where the liquid is exposed to a pulsating or alternating magnetic field^{12,13} or passed through a static magnetic field.¹⁴

In the present work, effects of the static magnetic field on the redox potential intensification and the stability of selected reducing agents (sodium dithionite and glucose) and oxidizing agents (hydrogen peroxide and sodium hypochlorite) are investigated in order to lower the process temperature, to improve the quality of process-

ing, and to reduce the consumption of environmental polluting chemicals. Redox potential measurements of these water solutions with a relevant quantity of sodium hydroxide were monitored after their exposure to the magnetic field and compared with the results for untreated ones.

EXPERIMENTAL

Preparation of Solutions

For all experiments, solutions of reduction and oxidation agents of different concentrations were prepared. The chemicals were obtained from Sigma-Aldrich Company. Two reduction agents, sodium dithionite ($\text{Na}_2\text{S}_2\text{O}_4$) and glucose ($\text{C}_6\text{H}_{12}\text{O}_6$), and two oxidizing agents, hydrogen peroxide (H_2O_2 , $w = 30\%$) and sodium chlorate (NaClO), with adequate amounts of NaOH were dissolved in water with the final pH in the range from 12 to 13 in reducing solutions and from 10 to 11 in oxidizing solutions.

Magnetic Treatment

Each solution was magnetically treated by passing it through a static magnetic field (Figure 3) at a flow velocity 1 m s^{-1} (1.2 L min^{-1}) at room temperature (20°C). Solutions were conducted through a cylindrical glass pipe (0.5 cm diameter) inserted into the magnetic device. The applied magnetic device consists of alternately arranged permanent magnets, yielding a magnetic field with three maximums of density 0.7 and 0.9 V s m^{-2} (Figure 4). The density curve along the axis at the edge of the gap is only slightly lower than the curve along the centre-axis.

Additionally, sodium dithionite and hydrogen peroxide solutions were circulated through the device two or three-times so as to observe the influence of retention time of the solution in the magnetic field on ORP.

Redox Potential

– Standardization and Cleaning of Redox Electrode

Chromic acid (5 g potassium dichromate dissolved in 500 ml sulphuric acid) was used for cleaning the electrode and

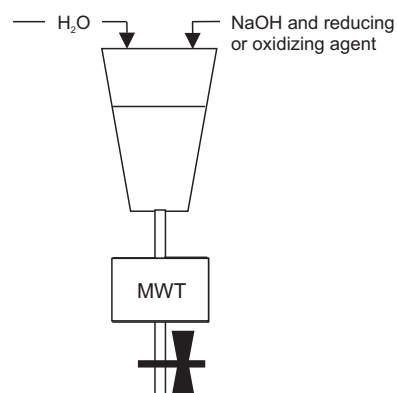


Figure 3. Scheme of the magnetic exposure procedure.

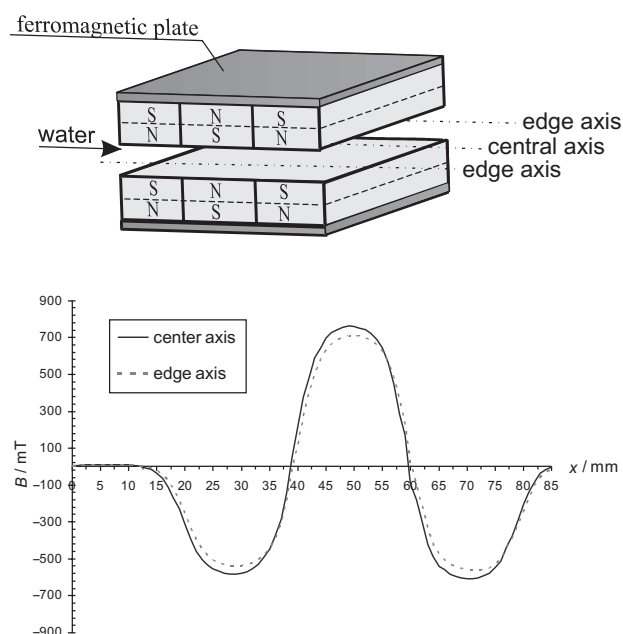


Figure 4. Arrangement of magnets in the MWT device and the distribution of magnetic field density in the gap.

standard ZoBell's solution (3 mmol dm⁻³ potassium iron(II) cyanide and 0.02 mol dm⁻³ potassium iron(III) cyanide in 0.1 M KCl) was used for standardization of the measuring instrument at 25 °C before each measurement.¹⁵

– Redox Potential Measurements

After completion of the magnetic treatment, the solution was immediately heated for 10 min under stirring in the cell without air (Figure 5). Heating was completed at 60 °C for reducing solutions and at 40 °C for oxidizing solutions. Redox potential was then monitored each minute for 30–75 minutes by a platinum electrode *vs.* the Ag/AgCl reference electrode with KCl electrolyte, connected to a pH meter (Mettler Toledo MA 235, pH/Ion Analyzer). Measurements were repeated for untreated solutions (reference), starting at the same temperature and time point after the components were mixed into the solution, as it was done in the case of

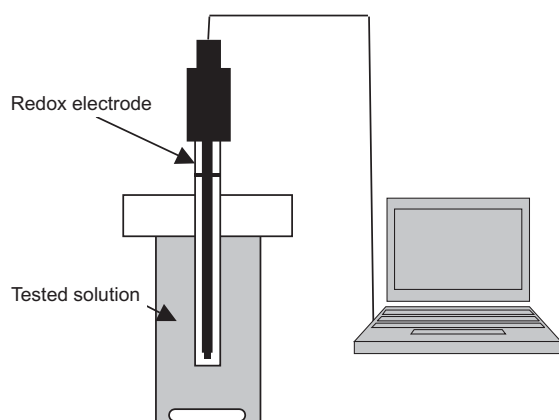


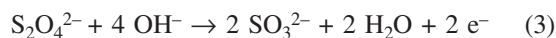
Figure 5. Test cell scheme.

magnetic treatment. Average values of three measurements (made with fresh solutions) are given as the final results.

RESULTS AND DISCUSSION

Effect of Magnetic Field on the Sodium Dithionite Solution

The importance of sodium dithionite in industry lies in its powerful reducing capacity (due to the S–S bond and the oxidation state of sulphur in the compound), which allows, for example, vat dyes to be reduced at room temperature.²¹ Solution of Na₂S₂O₄ is very unstable and requires special handling. Since it reacts with aqueous as well as with atmospheric oxygen (Eq. 2), sodium hydroxide was added to water before Na₂S₂O₄ addition. The oxidation reaction (Eq. 3) took part:



The redox couple that defines the platinum electrode potential is:

$$E = E^\circ - \frac{RT}{nF} \ln \frac{[\text{S}_2\text{O}_4^{2-}][\text{OH}^-]^4}{[\text{SO}_3^{2-}]^2} \quad (4)$$

Figure 6 represents the redox potential curves for the reference and the magnetically treated solutions of Na₂S₂O₄ with NaOH at different agent concentrations. Na₂S₂O₄ has strong reducing capacity due to its electron donating ability, which means that the surface of the platinum measuring electrode (Pt²⁺ + 2 e⁻ → Pt) gains a negative charge. With increasing time and concentration, the electrode generates more negative voltage. As can be seen from the curves, there is no significant difference in reduction ability or reaction rate to reach maximum potential. After 20 minutes, the reduction potential of magnetically treated solutions became stable and remained at this level for over an hour (data not shown) compared to magnetically untreated solutions, where the redox potential value started losing its negative redox value. Besides, deviation of reduction potential values of magnetically treated samples is obviously lower (Figure 7) compared to magnetically untreated solutions, indicating again better stability and reproducible ability of these magnetically treated samples. Figure 8 represents the redox potential curves for the reference and the one, two and three times magnetically treated solutions of 1 g L⁻¹ Na₂S₂O₄ with NaOH. The results show that the one time treatment is optimal, while the results of two and three time treatments are actually poorer than the reference solution. Loss of negative redox potential can be explained by the reaction (oxidation) of Na₂S₂O₄ with O₂ from the air; the same solution runs through the magnet several times and the air contact is increased.

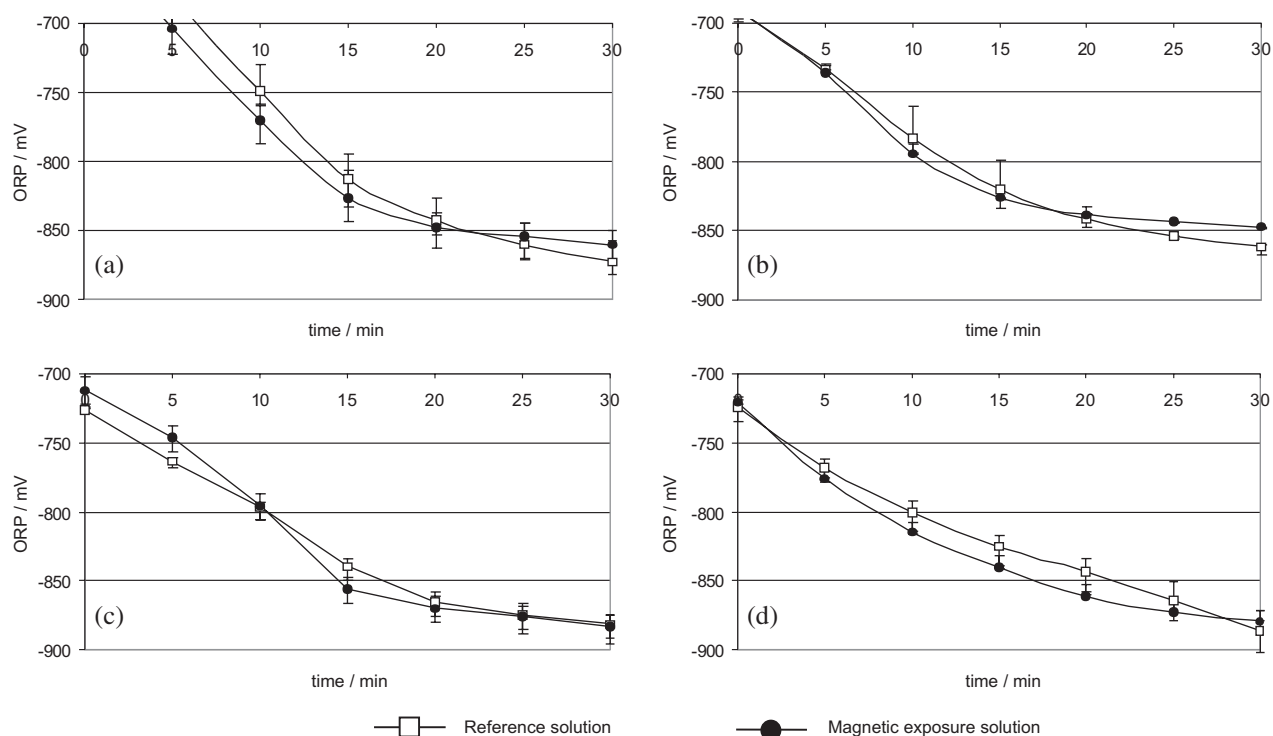


Figure 6. The magnetic field effect on the reduction potential of $\text{Na}_2\text{S}_2\text{O}_4$ water solutions ((a) 1 g/L, (b) 2 g/L, (c) 3 g/L, (d) 5 g/L $\text{Na}_2\text{S}_2\text{O}_4$) with NaOH (4.7 g/L, pH = 12–13) at 60 °C.

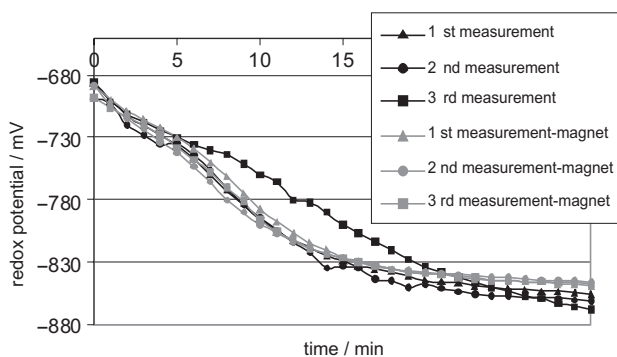


Figure 7. Effect of the magnetic field on stabilization of reduction potential of 2 g/L $\text{Na}_2\text{S}_2\text{O}_4$ water solutions with NaOH (4.7 g/L, pH = 12–13) at 60 °C.

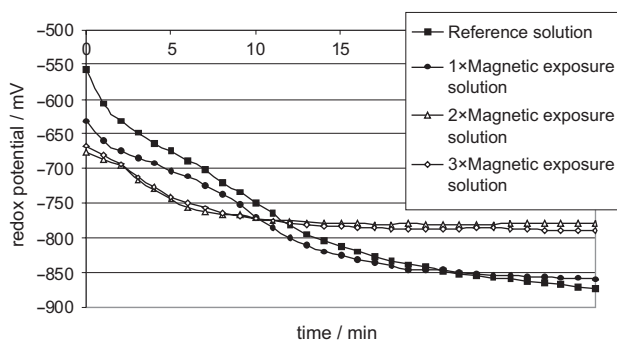


Figure 8. Effect of 1×, 2×, 3× magnetic field treatments on the reduction potential of 1 g/L $\text{Na}_2\text{S}_2\text{O}_4$ water solutions with NaOH (4.7 g/L, pH = 12–13) at 60 °C.

Effect of the Magnetic Field on Glucose Solution

Glucose (Figure 9), one of the most important biocompatible, biodegradable and non-toxic naturally occurring molecules, is known as a very good reducing agent, which is used in textile processes for dyeing with sulphur dyes and for bleaching of cotton fabrics.²² Glucose is a hexose – a monosaccharide containing six carbon atoms. Glucose is an aldehyde (contains a $-\text{CHO}$ group). Five carbons plus an oxygen atom form a loop called a »pyranose ring«, the most stable form of six-carbon aldoses. In this ring, each carbon is linked to hydroxyl and hydrogen side groups, with the exception of the fifth atom, which links to a 6th carbon atom outside the ring, forming the CH_2OH group. It exists in two modifications (α and β). Sugar crystallizes from aqueous solutions at temperatures below 50 °C as α -D-glucose monohydrate, which has the melting point of 80 °C. At temperatures above 50 °C but below 115 °C, the stable form is anhydrous α -glucose with the structure shown in Figure 9. Glucose undergoes a complex degradation sequence in alkaline solutions and the reducing effect of glucose is known to be associated with the degradation intermediate. In alkaline media, a complex sequence of degradation steps occurs, with dehydrated intermediates with extended π -systems being the most likely redox active reducing agents.^{23,24} The redox couple in glucose solution that defines the potential of platinum electrode cannot be determined because reducing intermediates are unknown.

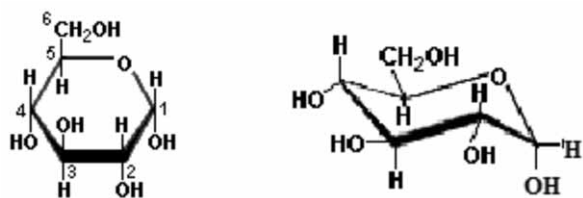


Figure 9. Chemical structure of α -D-glucose and α -D-glucose, chair form.

Glucose is highly sensitive to temperature; at high temperatures, glucose acts as a very good reducing agent. The subsidizing factors, which contribute to its reducing ability, are high alkalinity and polysulphide addition.²⁵ Since the reduction potential of sulphur dyes lies between -400 mV and -500 mV *vs.* Ag/AgCl, all measurements were carried out at 60 °C, the lowest temperature needed to reach a sufficient reducing potential. At alkaline pH and at around 60 °C and higher, glucose decomposes into reactive fragments, which have a high reduction capability. Figure 10 represents the curves of the reference and magnetically treated solutions of glucose with NaOH at 60 °C. It can be seen that magnetic treatment yielded up to 10 % of reducing ability and shortened the time of reaching it in dependence on the glucose concentration used. Anyhow, the concentrations

used (2 – 5 g L^{-1}) were not significantly different in the redox potential value; regardless of the glucose concentration used, the maximum reduction potential of around -600 mV *vs.* Ag/AgCl was reached in *ca.* 30 minutes at all concentrations except at 4 g L^{-1} glucose where it was reached around -700 mV *vs.* Ag/AgCl in 20 minutes. Figure 11 shows measurements of the reference, the magnetically treated solution of glucose with NaOH and the separate magnetic treatment of NaOH solution and glucose solution at 60 °C. There is no significant difference in redox potential between the separate magnetic treatment of NaOH and glucose solutions and magnetically treated solution of glucose with NaOH.

Effect of the Magnetic Field on Hydrogen Peroxide Solution

Hydrogen peroxide has a wide spectrum of applications not only in textile but also in paper production, manufacturing or processing of foods, minerals, petrochemicals, wastewater applications and medicine. Hydrogen peroxide is used with NaOH to produce a high pH that is necessary to produce the active perhydroxyl ion, HOO^- .^{25,26}

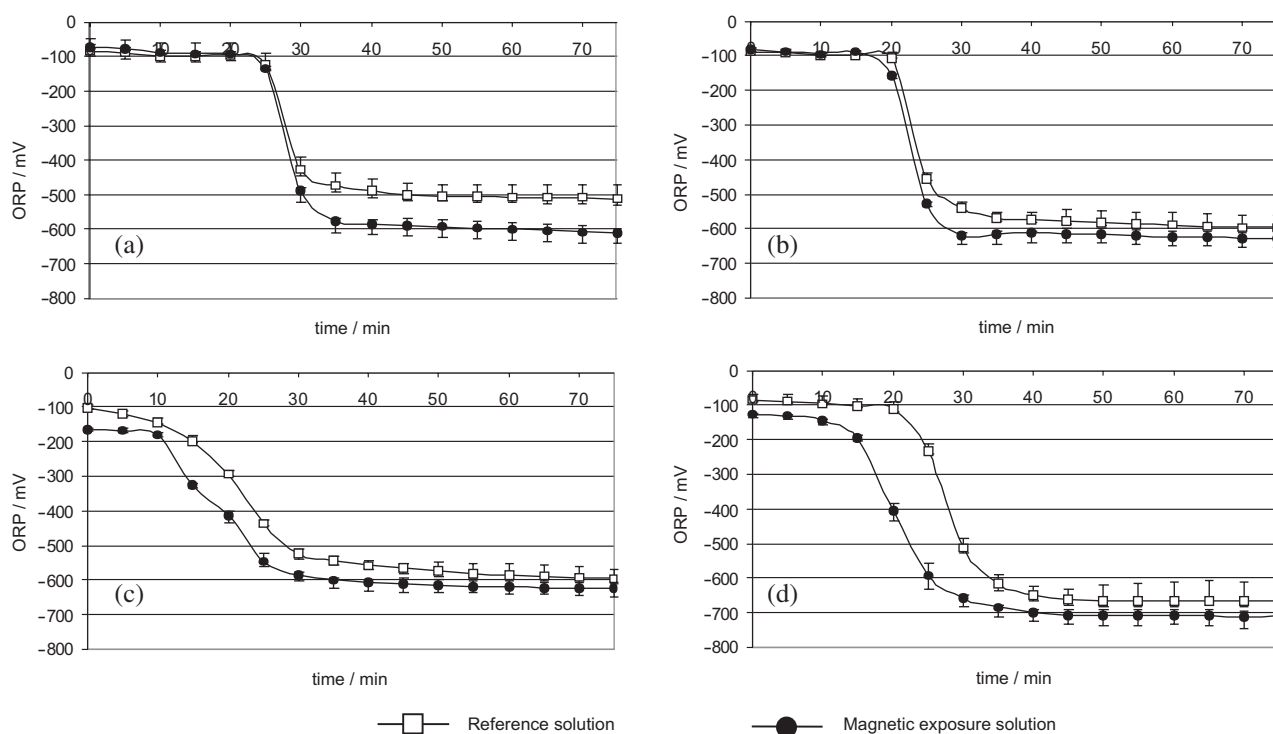
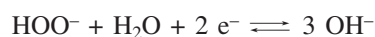
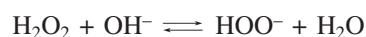


Figure 10. Effect of the magnetic field on reduction potential of $C_6H_{12}O_6$ water solutions ((a) 2 g/L, (b) 3 g/L, (c) 4 g/L, (d) 5 g/L $C_6H_{12}O_6$) with NaOH (4.7 g/L, pH = 12 – 13) at 60 °C.

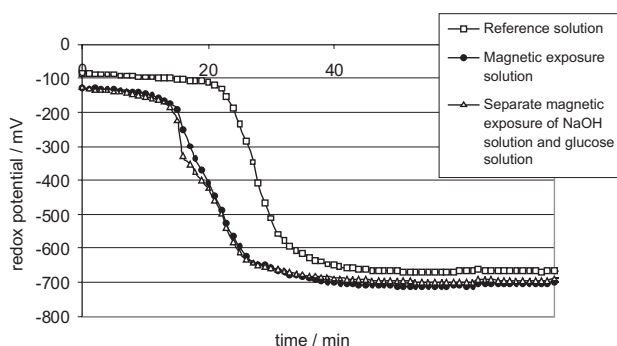


Figure 11. Effect of the magnetic field on reduction potential of 4 g/L $C_6H_{12}O_6$ water solutions with NaOH (4.7 g/L, pH = 12–13) and separate magnetic effect on the NaOH solution and glucose solution at 60 °C.

The redox couple that defines the platinum electrode potential is:

$$E = E^\circ - \frac{RT}{nF} \ln \frac{[OH^-]^2}{[H_2O_2]} \quad (6)$$

For bleaching and oxidation (vat dyeing) processes, temperatures around 40 °C are quite usual and can be carried out with great time saving, so the measurements were performed at that temperature.²⁷ H_2O_2 has oxidation capacity owing to its ability of accepting electrons, which reverse the reaction on the platinum measuring electrode. With increasing concentration, the electrode generates increasingly higher positive voltage. With increasing time, the

redox potential was not increased, which can be explained by the H_2O_2 reaction in water. Hydrogen peroxide always decomposes (disproportionates) exothermically into water and oxygen gas spontaneously: $2 H_2O_2 \rightarrow 2 H_2O + O_2$, so it loses its oxidizing capability with time. As it is seen from Figure 12, the magnetic treatment raises the curve, yielding about 10–20 % higher oxidation ability. This shift increases with increasing the H_2O_2 concentration, reaches its maximum (at around 4 mL L^{-1}) and remains constant for higher concentrations. Figure 13 represents the redox potential curves for the reference and the one, two and three times magnetically treated solutions of 2 mL L^{-1} H_2O_2 with NaOH. The results are the same as in the case of $Na_2S_2O_4$; the one time treatment is optimal, while two and three time treatments decreased the positive redox value. As mentioned above, hydrogen peroxide always decomposes in water to H_2O and oxygen, which was accelerated by additional solution decanting.

Effect of the Magnetic Field on Sodium Hypochlorite Solution

Sodium hypochlorite is a powerful oxidizing agent that is extensively used in agriculture and chemical industries and, as already mentioned, in the textile industry for surface purification (to remove stains from clothes at room temperature), for bleaching, and vat dyeing. Sodium hypochlorite is a salt of hypochlorite acid, HClO. Sodium hypochlorite solution is a light yellow green transparent liquid. In water, it partially splits into the sodium cation

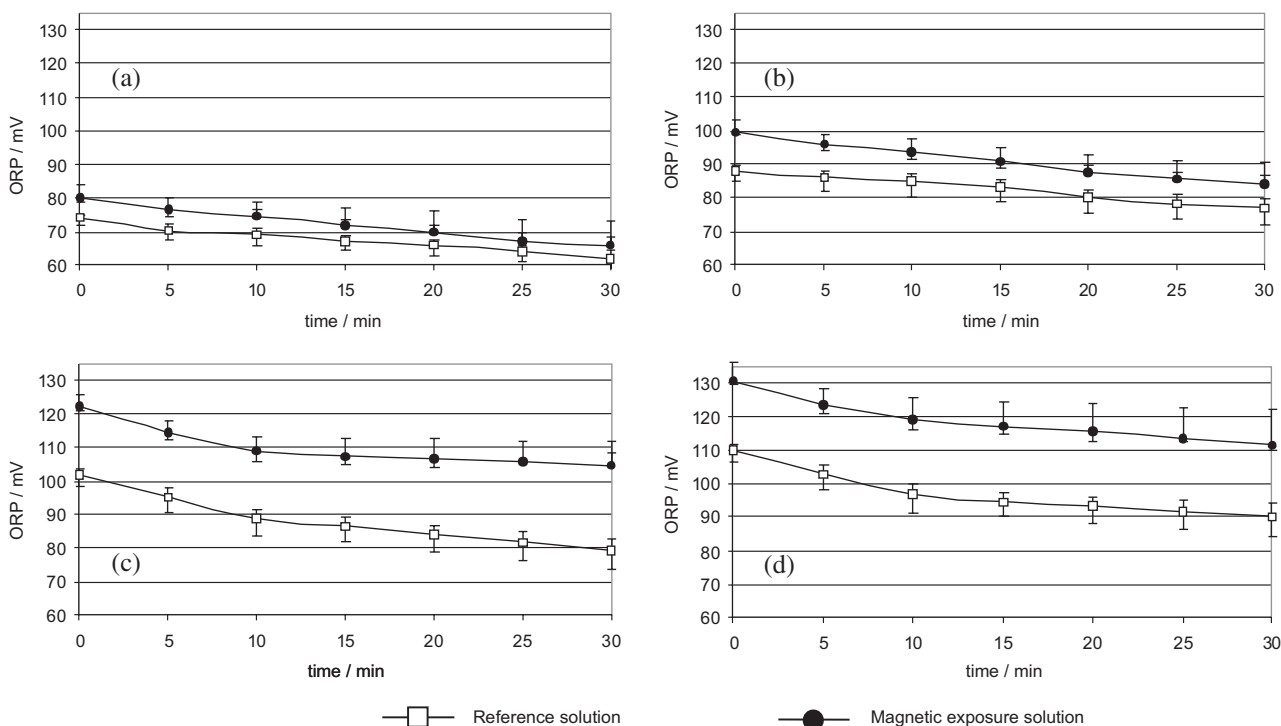


Figure 12. Effect of the magnetic field on oxidation potential of H_2O_2 solutions ((a) 1 mL/L, (b) 2 mL/L, (c) 4 mL/L, (d) 6 mL/L H_2O_2 , w = 30 %) in the presence of NaOH (0.235 g/L, pH = 10–11) at 40 °C.

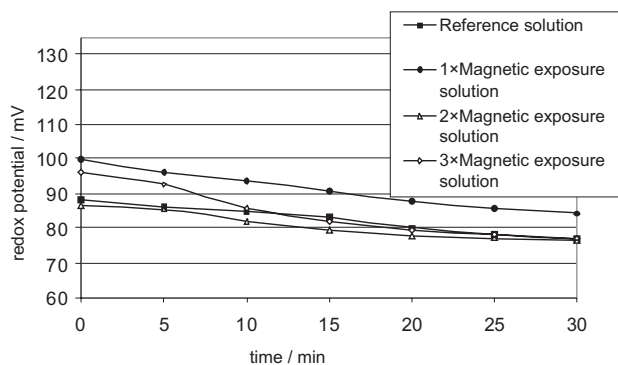
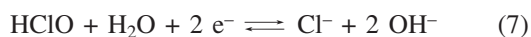


Figure 13. Effect of 1×, 2×, 3× magnetic field treatments on oxidation potential of 2 mL/L H₂O₂ (w = 30 %) solutions in the presence of NaOH (0.235 g/L, pH = 10–11) at 40 °C.

Na⁺ and the hypochlorite anion ClO⁻, while a substantial portion hydrolyzes into sodium hydroxide and hypochlorite acid. The oxidizing power of the latter and of the hypochlorite anion causes the bleaching effect:²⁸



The redox couple that defines the platinum electrode potential is:

$$E = E^\circ - \frac{RT}{nF} \ln \frac{[\text{Cl}^-][\text{OH}^-]^2}{[\text{ClO}^-]} \quad (8)$$

Figure 14 represents the curves for treated and untreated solutions of NaClO in the presence of NaOH at

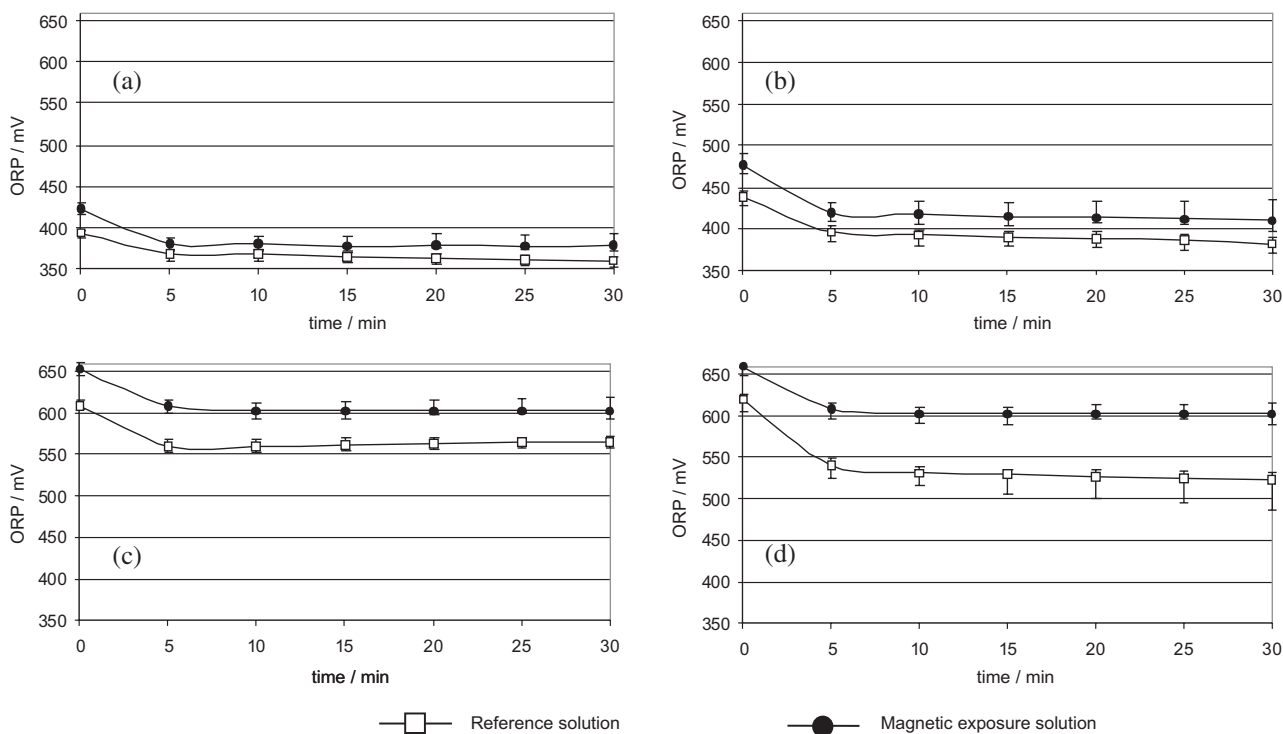


Figure 14. Effect of the magnetic field on oxidation potential of NaClO solutions ((a) 1 mL/L, (b) 2 mL/L, (c) 3 mL/L, (d) 4 mL/L NaClO and pH of 10–11) in the presence of NaOH (0.235 g/L, pH = 10–11) at 40 °C.

40 °C. As can be seen from the measurement curves also in these cases, the magnetic treatment increased the oxidation potential. Similarly, the increase was proportional to the concentration of the oxidizing agent.

CONCLUSIONS

The redox potential of different reduction and oxidation agents was monitored after the exposure of their solutions (containing NaOH) one, two and three times to the static magnetic field, and compared to the measurement results for untreated samples. The one time magnetic treatment significantly increased both reduction and oxidation abilities of the agents.

Special care was taken to conduct the experimental procedure in exactly the same way for magnetically treated and untreated samples. Untreated samples were mixed in the reservoir and also passed through the pipe, which was without magnets, to provide the same hydrodynamic conditions in the mixing procedure.

Results of the experiments show that the magnetic treatment raises both the reducing ability of glucose and the oxidation ability of hydrogen peroxide and sodium hypochlorite, while there was no significant change in the oxidation potential of Na₂S₂O₄ solution. As regards the mechanism of magnetic field effect, an explanation may be searched in H-bond distortion in the hydration shell of dissolved chemicals. In all three cases, chemicals have

–OH groups, which tend to form H-bonds in water solution, as it was demonstrated. The best result among these chemicals was that for H₂O₂, which is very similar to H₂O. Accordingly, the increased reduction/oxidation potential might be due to the magnetically increased deprotonization of the agents, but to confirm this, more theoretical and experimental research has to be done. Anyhow, the increase in redox potential due to magnetization of water treatment promises some technological and economical benefits for textile technological processes as well as for other fields of chemistry, e.g., for syntheses of pharmaceuticals, dyes, paints, and a multitude of other important products.

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SAŽETAK

Utjecaj magnetskog polja na redoks potencijal redukcijskih i oksidacijskih spojeva

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Redoks potencijali vodenih otopina dvaju redukcijskih (natrijev ditionat i glukoza) i dvaju oksidacijskih (vodikov peroksid i natrijev hipoklorit) spojeva mjereni su pri različitim koncentracijama i različitim temperaturama 30–75 minuta poslije izlaganja otopina statičnom magnetskom polju gustoće toka 0,9 V s m⁻². Otopine su polju izlagane ili samo jednom (glukoza i natrijev hipoklorit) ili jednom, dvaput i triput (natrijev ditionat i vodikov peroksid). Cilj je istraživanja predložiti poboljšanje redukcijske, odnosno oksidacijske sposobnosti odabranih spojeva koji se koriste u obradi tekstilnih vlakana, prvenstveno za izbjeljivanje i bojenje. Rezultati eksperimenata pokazuju da se magnetskom obradom otopina povećavaju redukcijska sposobnost glukoze i oksidacijska sposobnost vodikova peroksida i natrijeva hipoklorita, što bi moglo biti tehnološki i ekonomski korisno tekstilnoj industriji, a zanimljivo ostalim granama kemije.