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Synthesis of a novel visible light-driven and magnetically separable photocatalyst based on $BiFeO_3$-$YFeO_3$ type II heterojunction

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Abstract. Nanocomposites based on the $BiFeO_3$-$YFeO_3$ system with different phase ratios have been synthesized via the solution combustion route. The nominal compositions were set as $x$$BiFeO_3$-(1-$x$)$YFeO_3$ (where x = 0.1, 0.3, 0.4, 0.5, 0.6, 0.7, and 0.9), labeled from BYFO-01 to BYFO-09. Glycine was used as a reducing agent for the combustion reaction. The average crystallite size of the obtained materials was in the range of 25–90 nm. A study of the magnetic characteristics and photocatalytic activity of the samples showed that variations in their phase composition significantly enhance both total magnetization and the photodegradation of Rhodamine B dye. All samples exhibit magnetic ordering at room temperature and increased magnetization compared to pure bismuth and yttrium orthoferrites. It was found that the BYFO-04 nanocomposite possesses enhanced photocatalytic activity, which was attributed to the approximately 1:1 orthoferrite phase ratio. The BYFO-0.5 and BYFO-0.7 nanocomposites showed an improved magnetic response, with specific and saturation magnetization values of approximately 8 emu/g at a field of 4 kOe. The increased magnetization of these samples was explained by their phase composition, which can be technologically controlled. The discovered properties of the synthesized materials are promising for the effective separation of photocatalysts from the reaction medium. The BYFO-0.4 sample photodegraded about 40% of the rhodamine B dye and 80% of methyl violet within 180 minutes of irradiation with visible light. The easy synthesis method, combined with magnetic and photocatalytic responses and environmentally benign iron sources, makes $BiFeO_3$-$YFeO_3$ nanocomposites promising materials for photocatalytic systems and memory recording applications.

Keywords: orthoferrites, nanoparticles, combustion method, magnetic properties, photocatalytic activity

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1. Introduction

Recent research of multiferroics has focused on orthoferrites $AFeO_3$ (A – Bi, Y) due to their promising magnetic and electrical characteristics, and photocatalytic activity [1–16]. At present, orthoferrites $BiFeO_3$ and $YFeO_3$ are used as materials for magnetic storage devices [1,2,4,8,9], gas sensors [2,4], photocatalytic systems [3,5–7,9,10,17,18], etc.

Bismuth orthoferrite ($BiFeO_3$) is popular magnetoelectric material because it exhibits multiferroic properties at room temperature and above it. $BiFeO_3$ has the Neel temperature of T_N≈360°C and Curie temperature of T_C≈830°C [9]. Below T_C, bismuth orthoferrite exists in the form of an α-phase with a rhombohedral perovskite-like crystal structure (*R3c* space group) [19]. This material combines the properties of ferroelasticity, antiferromagnetism and ferroelectricity [2,9]. The magnetic nature of $BiFeO_3$ is described as *G*-type antiferromagnetic order of Fe^{3+} ions with a space-modulated cycloid spin structure having a period λ_c=62±2 nm [2]. $BiFeO_3$ nanocrystals exhibit weak ferromagnetism arising from the canting of the antiferromagnetic Fe sublattices and the appearance of the Dzyaloshinskii-Moriya effect [9,13].

Yttrium orthoferrite ($YFeO_3$) is an attractive material due to its excellent magneto-optical properties and extraordinary domain wall motion [20]. In addition, the coupling of magnetic and ferroelectric ordering in $YFeO_3$ makes it us to consider this compound as a promising multiferroic [21].

One of the ways to improve the performance of the orthoferrites is to create nanocomposite materials, i.e., of systems that include two or more components [13–16,22–29]. Recently, the development of nanocomposites has become a key area in the research of advanced multiferroics. Of particular interest are materials with magnetic order at room temperature. Moreover, nanocomposites can have improved photocatalytic activity due to the formation of new active centers. The synthesis and properties of nanocomposite materials based on bismuth and yttrium orthoferrites with different phase compositions have been described in [14,24,27–31]. Most researchers indicate that composite materials based on orthoferrites have improved magnetic and photocatalytic responses and can be synthesized using «soft» chemical methods. Nanomaterials based on bismuth and yttrium orthoferrites are photocatalysts whose performance can be enhanced by doping [22]. These materials have proven to be very useful for the degradation of dyes under visible light irradiation among other photocatalysts.

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Solution combustion synthesis is an effective method for the synthesis of oxide nanoparticles and nanocomposites since it is an energy- and time-saving method that does not require expensive equipment [13,14,32–40]. Currently, this technique is actively used for the synthesis of orthoferrites (e.g. [3,13,14,27,30,32,39–46]). Such conditions of synthesis as the type of organic fuel and the ratio of fuel to oxidizer strongly affect the formation and properties of oxide nanomaterials [39,41,42,45,46], including $BiFeO_3$ and $YFeO_3$.

The available data on the properties of the $Bi_{1-x}Y_xFeO_3/Y_{1-x}Bi_xFeO_3$ materials are fragmentary and apply only to some compositions. Materials based on the $BiFeO_3$-$YFeO_3$ system are presented in the literature, mainly as solid solutions of the $Bi_{1-x}Y_xFeO_3/Y_{1-x}Bi_xFeO_3$ type with a narrow range of $x<0.3$ [5,47–51]. It is noted that with increasing doping, the magnetic response and photocatalytic activity of the $Bi_{1-x}Y_xFeO_3$ materials improve [47,48,51]. The successful synthesis of phases in the range $0<x<1$ has apparently been reported only in [52], but the functional properties of most samples have not been studied. There are no publications devoted to the magnetic study of nanocomposites $BiFeO_3$-$YFeO_3$. The increased photocatalytic activity of Y-doped nanocomposites is reported in [5,51]. Based on the data on doped materials and pure orthoferrites, we hypothesize that the $BiFeO_3$-$YFeO_3$ system may yield new materials with magnetic order at room temperature and enhanced magnetic and photocatalytic responses.

The aim of this study was to synthesize $BiFeO_3$–$YFeO_3$ nanocomposites via glycine-nitrate combustion synthesis and analyze the obtained materials in terms of their magnetic and photocatalytic applications.

2. Materials and methods

2.1 Materials and reagents

Nanocomposites based on the $BiFeO_3$–$YFeO_3$ system were synthesized via the glycine-nitrate combustion method. Crystallohydrates of metal nitrates $Bi(NO_3)_3 \cdot 5H_2O$, $Y(NO_3)_3 \cdot 6H_2O$, $Fe(NO_3)_3 \cdot 9H_2O$, and glycine $C_2H_5NO_2$ of p.a. purity grade were used as initial reagents. Metal nitrates were mixed in a proportion corresponding to the nominal compositions of $x$$BiFeO_3$-$(1-x)$$YFeO_3$ with x = 0.1, 0.3, 0.4, 0.5, 0.6, 0.7, and 0.9.

2.2 Materials synthesis procedure

The amount of glycine used as organic fuel corresponded to a glycine-nitrate (*G/N*) ratio of 0.55. Metal nitrates and glycine were dissolved in 100 ml of 1M HNO_3 aqueous solution with constant stirring and heating. The obtained solutions were heated until the water evaporated and the reaction mixtures spontaneously combusted, resulting in the formation of

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brown powders. The combustion products were sequentially calcined in the temperature range of 350–750 °C for 1 hour at each stage.

Thus, reference samples of $BiFeO_3$ (BFO) and $YFeO_3$ (YFO) were synthesized, as well as a series of xBiFeO$_3$-(1-x)YFeO$_3$ (x = 0.1, 0.3, 0.4, 0.5, 0.6, 0.7, and 0.9) samples. The mixes Bi-Y orthoferrite samples were named according to Y content - from BYFO-01 to BYFO-09. The prepared samples were characterized by following physicochemical methods.

2.3 Materials characterization

The morphology of the samples was studied by scanning electron microscopy (FEI Quanta 200 SEM with the EDAX attachment), and the elemental composition of the samples was checked by energy dispersive X-ray analysis.

The powder X-ray diffraction (XRD) patterns of the samples was measured at room temperature by using CuKα X-ray source (Rigaku SmartLab 3) for the Bragg angle (2θ) range of 20-70°. To calculate unit cell parameters, the SmartLab software package was used.

A Wissel spectrometer with a ^{57}Co source in a rhodium matrix was used for the Mössbauer examination of the sample at room temperature (isomeric shifts *IS* are given relative to the *IS* of α-Fe). Spectra fittings were made with the Mossfit software.

Magnetic measurements were performed with the Physical Properties Materials System (PPMS) provided by the Quantum Design using vibrating sample magnetometer option. At room temperature, the dependences of the specific magnetization M on the field H were studied. For single-phase samples, they were measured up to 40 kOe, for nanocomposites - up to 4 kOe.

The photocatalytic activity was assessed by photodegradation of the Rhodamine B dye (RdB) under the action of visible light. 30 mg of the photocatalyst was added to a solution of 10 mg/L RdB, and the resulting mixture was treated with ultrasound for 3 min to achieve maximum dispersion. To increase the photocatalytic activity (photo-Fenton-like mode), 1 mL of 3% H_2O_2 was added. Before irradiation, the mixture was stirred for 60 minutes without light, and then the degree of dye adsorption and desorption on the catalyst surface was determined.

The visible light source was a 300 W Osram Vita-lux lamp with a UV filter. The irradiation intensity in all experiments was 15 mW/cm^2. The probes were taken every 30 minutes, and the powders were separated from them by a centrifuge. The rate of photocatalytic degradation of RhB was estimated from the change with time in the absorption spectrum of the solutions. The maximum absorption of RhB is at a wavelength of 550 nm. The absorption spectrum of the solutions was determined on an EMC-11-UV spectrophotometer.

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The light absorption ability of the samples was investigated by diffuse reflectance spectra (DRS) in the UV-visible region recorded at room temperature in the range of 400–800 nm using an AvaSphere-30-Refl integrating sphere.

The band gap energy (E_g) was calculated from the plot of the Kubelka-Munk function:

$$F(R) = \frac{(1-R)^n}{2R} = \frac{K}{S} \quad (1)$$

where K is the molar absorption coefficient, S is the scattering factor, and R is the reflectance of the material. The n value is determined by the nature of the sample (n = 2 for direct allowed transitions and n = ½ for indirect allowed transitions).

For statistical analysis of the entire volume of the obtained data, the Pearson matrix was used. For the machine analysis of the dependence of the functional characteristics of $BiFeO_3$–$YFeO_3$ nanocomposites on the composition, variables reflecting the content of bismuth and yttrium were used. The experimentally measured parameters characterizing the photocatalytic properties (reaction rate constant k, min^{-1}; degree of adsorption; time to reach 10% degradation $t_{10\%}$, min; removal efficiency RE; final dye concentration C_{final}, g/L; amount of reacted substance $C_{reacted}$, g/L) and magnetic parameters (excess iron Fe_{excess}; magnetization M, emu/g at 4 kOe; residual magnetization M_r, emu/g; coercive force Hc, kOe) were used as response variables. Eight models were used to describe the relationships between composition and functional properties: a classical polynomial regression model and seven machine learning algorithms: CatBoost, Gaussian process regression (GPR), ElasticNet-poly, ElasticNet, eXGBoost, eLasso, and a support vector machine (SVR). Model evaluation was performed using the coefficient of determination.

3. Results and discussion

3.1. Energy-dispersive X-ray (EDX) spectroscopy and scanning electron microscopy (SEM)

The morphology and elemental composition were investigated by SEM/EDXS. The EDX spectra of two typical samples (BYFO-03-09) are shown in Fig. S1 and indicate that the prepared materials contain only the desired Bi, Y, Fe, and O elements. Elemental analysis of BFO and YFO samples showed that, the metals ratio in which were 1:1, i.e., it corresponded to the $BiFeO_3$ and $YFeO_3$ stoichiometry (see Table S1).

Additionally, samples BFO, BYFO-01, and BYFO-09 have average element ratios of Bi/Y/Fe that are fairly close to the nominal values. An excess of iron (d) was detected in the

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BYFO-03-07 samples that had the highest concentration of iron-containing impurities (Fig. S1) (see Table S2).

Fig. S2 presents SEM images of the samples. It can be seen that the BFO morphology is a network of sintered grains with a rhombic-like shape. With changes in phase and elemental composition of the materials, the grain morphology becomes less ordered. Doped samples have porous grains with cavities that were formed due to the rapid release of the combustion gases.

The grain size was determined by measuring a substantial number of grains in SEM images. Y-doping leads to a decrease in particle size and a change in morphology. Thus, samples BYFO-01-04 consist of porous agglomerates of elongated particles, and their micrographs indicate that the grain size is reduced three times (Table S1).

Yttrium ions are less mobile than Bi^{3+} ions, and they slow down the solid-state reaction during sequential heat treatment of samples, preventing grain growth. The synthesis temperature of these materials was 750 °C, compared to 550 °C previously used for the glycine-nitrate synthesis of pure $BiFeO_3$ [46].

Many dopants were reported to suppress grain growth, and other authors have also assumed that doped $BiFeO_3$ may require slightly higher sintering temperatures. A similar particle morphology was observed in [48], with yttrium doping up to 10%. With a subsequent increase in yttrium content, the particles become flake-shaped and aggregate into disordered porous particles, the size of which increases with increasing Y (see Fig. 2, Table 2, samples BYFO-06-09). The pure YFO sample consists of highly aggregated flake-like particles with an average grain size of 2.1 μm, which is the maximum for all series of samples.

3.2. Powder X-ray diffraction (PXRD)

The selection of nanocomposite synthesis regimes was based on previously data on pure $BiFeO_3$ with enhanced magnetic and photocatalytic characteristics [37]. The samples were sequentially heat-treated in the range of 350–750°C. After annealing at 750°C, the samples contained minimal amounts of impurity phases.

Fig. 1 shows the XRD patterns of the samples after final heat treatment at 750 °C. The observed X-ray reflections of the main phase in the BFO and BYFO-01 samples correspond to bismuth orthoferrite $BiFeO_3$. The peaks in these PXRD patterns can be indexed to the rhombohedral, distorted perovskite-like structure (PDF #14-181). In the BYFO-09 and YFO samples, the main phase is perovskite-like yttrium orthoferrite. The reflections in their PXRD patterns correspond to orthorhombic phase $YFeO_3$ (PDF #8-150). The crystallite sizes of the main phases in these samples are given in Table 1. Obtained results are consistent with other

works [5,51], and indicate the formation of $Bi_{1-x}Y_xFeO_3/Y_{1-x}Bi_xFeO_3$ solid solutions at values of $x \leq 0.1$.

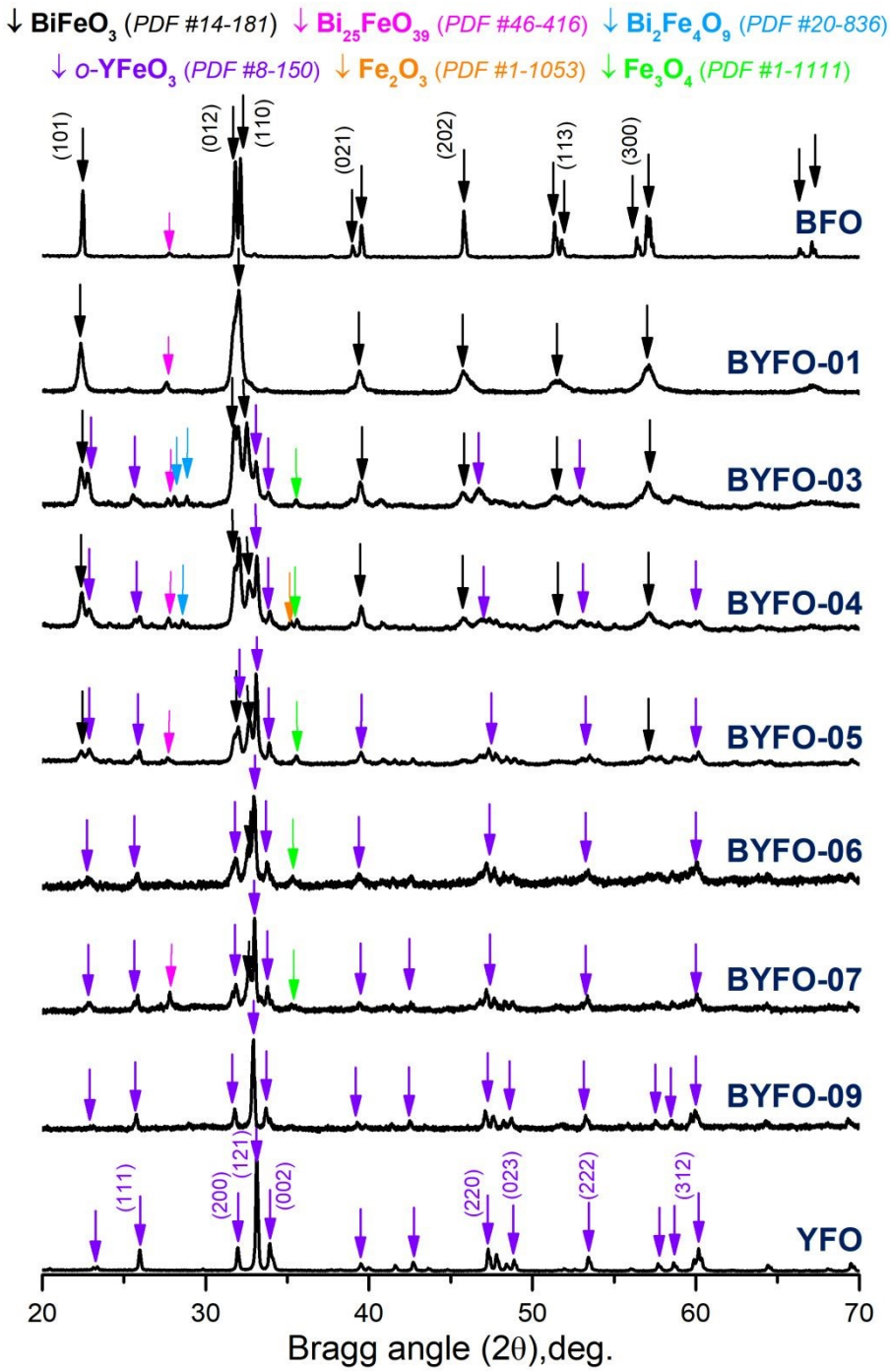

Fig. 1. PXRD patterns of the BFO-YFO samples after heat treatment at 750 °C.

The observed X-ray reflections for the BYFO-03-07 samples correspond to a mixture of the main phases, $BiFeO_3$ and $YFeO_3$. Along with these phases, almost all samples contain traces of bismuth ferrites with sillenite-like ($Bi_{25}FeO_{39}$) and mullite-like ($Bi_2Fe_4O_9$) structures. These composite materials likely contain small amounts of iron oxides. Specifically, the

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BYFO-03-07 samples contain a small amount of magnetite Fe_3O_4, which exhibits a weak characteristic (311) peak around 35.4°. Detailed information on the phase composition of the synthesized samples is presented in Fig. 2.

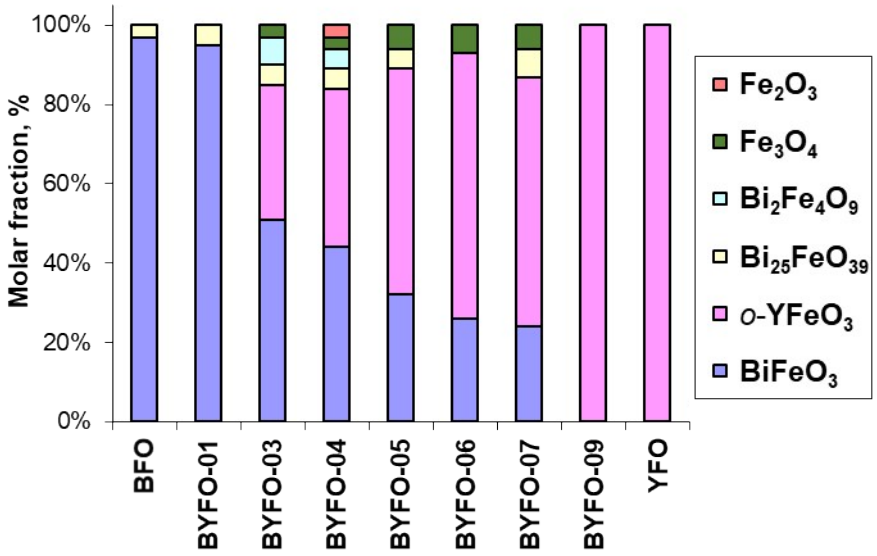

Fig. 2. Phase composition of the BFO-YFO samples

The causes of the formation of composites in the intermediate region of yttrium concentration may be the following: Firstly, the bismuth content decreases with increasing yttrium content in these materials, and besides, Bi_2O_3 evaporation begins above 700 °C. Therefore, an excess amount of bismuth nitrate in the initial composition might be required to avoid the formation of secondary phases.

According to [47], the appearance of these by-products could also be caused by lattice strain resulting from the ionic size mismatch between the host and substituent cations. The effective ionic radius of Y^{3+} (1.02 Å for coordination number $CN = 8$) is smaller than that of Bi^{3+} (1.17 Å in the same coordination) [53]. To further clarify the phase composition of the synthesized materials, the data from ^{57}Fe Mössbauer spectroscopy data are presented in the following subsection.

The average crystallite sizes (d) of the synthesized particles were estimated from the XRD patterns using the Scherrer equation and are given in Table 1. The calculation was performed using the most intense reflections of the primary phases, $BiFeO_3$ (012/110) and *o*-$YFeO_3$ (121/002). The average crystallite sizes vary depending on the phase composition, which may be due to spatial limitations on their growth. The estimated d values for the orthoferrites $BiFeO_3$ and $YFeO_3$ are 87 nm and 71 nm, respectively (see Table S1). The

crystallite size in the doped materials is smaller than in the pure orthoferrites' samples, as can be seen for samples BYFO-01 and BYFO-09. In addition, we did not observe significant differences in the crystallite size for the BYFO-01 and BYFO-09 samples or the composite samples. The lack of a clear dependence of *d* value on the sample composition may be caused by the overlap of diffraction lines from the main phases, making their precise separation difficult.

3.3. ^{57}Fe Mössbauer spectroscopy

^{57}Fe Mössbauer spectra of the BFO, BYFO-05, BYFO-09, and YFO samples are shown in Fig. 3. As determined from the spectra, the synthesized materials contain magnetically ordered phases at room temperature. The spectra of the BFO and YFO samples consist of sextets, with parameters characterizing the state of iron ions in the magnetically ordered $BiFeO_3$ and $YFeO_3$ phases, respectively [42,45,46]. The spectra of composite samples BYFO-05 and BYFO-09 represent a superposition of sextets and doublets. The parameters of the two doublets in these spectra correspond to octahedrally coordinated Fe^{3+} ions in superparamagnetic $BiFeO_3$ particles and paramagnetic (or non-magnetic) iron species.

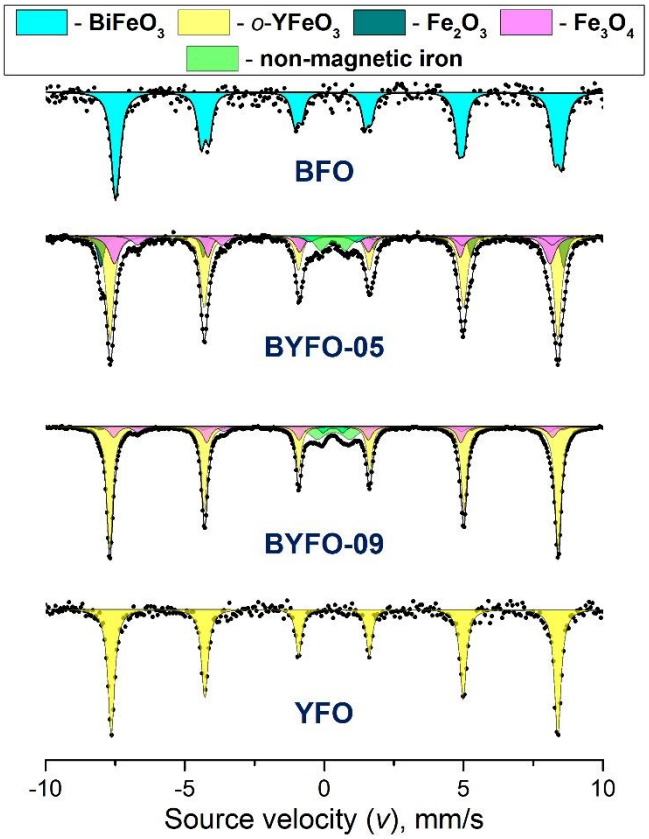

Fig. 3. ^{57}Fe Mössbauer RT-spectra of the BFO, BYFO-05, BYFO-09, and YFO samples.

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Comparing the results of the XRD and ^{57}Fe Mössbauer spectroscopy, it is clear that the BYFO-04-09 samples contain several iron-containing phases: orthoferrites $BiFeO_3$ and $YFeO_3$, Fe_2O_3 and Fe_3O_4 iron oxides, and a phase with non-magnetic iron. In the BYFO-05 and BYFO-09 samples, the majority of the iron atoms can be attributed to the magnetically ordered $YFeO_3$ phase, the content of which is higher in the BYFO-09 sample. Table S3 summarizes the spectral parameters of the primary phases in comparison with the literature data.

3.4. PPMS vibrational magnetometry

The magnetization (*M*) of the orthoferrite samples as a function of the magnetic field (*H*) at 300K is shown in Fig. 4a . The hysteresis loops were recorded at room temperature (RT). The presence of these hysteresis loops indicates that both BFO and YFO materials exhibit magnetic ordering at RT.

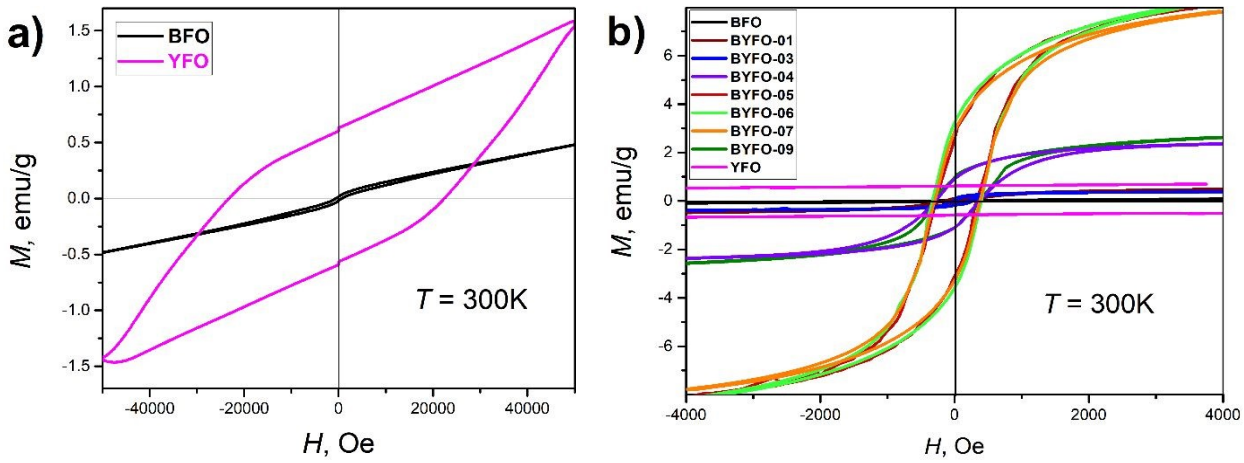

Fig. 4. RT magnetic hysteresis loops of pure BFO, YFO (a) and BYFO (b) samples.

Nanocrystalline $BiFeO_3$ has a narrow hysteresis loop and non-zero magnetization. The non-saturating behavior of the loop implies the presence of competing antiferromagnetic interactions in $BiFeO_3$ nanoparticles that have an ordered AFM core and uncompensated spins at the surface of nanocrystals [54]. Surface magnetization contributes to the total magnetic response of $BiFeO_3$ nanoparticles, and such materials can exhibit significantly higher magnetization compared to bulk samples [9]. In our case, the average size of $BiFeO_3$ crystallites is slightly larger than the period of the spin cycloid $d > \lambda_c$ (see Table S1), as a result of which this sample exhibits only a small magnetic response, which is consistent with other studies [4,9,39].

The YFO sample displays magnetic properties typical of nanocrystalline yttrium orthoferrite obtained by glycine-nitrate combustion. At RT, weak ferromagnetic behavior is observed, resulting from spin canting in the antiferromagnetically ordered Fe^{3+} sublattice, which is caused by Dzyaloshinskii-Moriya interactions. The magnetic hysteresis loop shows low remanent magnetization (M_r = 1.0 emu/g) and high coercivity (H_c = 23 kOe), indicating strong magnetic anisotropy. The plate-like morphology of the crystallites with an average size of 40 nm contributes to the magnetic hardness of the sample. These results confirm the presence of magnetic ordering at RT, consistent with previously reported data for nanocrystalline $YFeO_3$.

The magnetization curves $M(H)$ of the nanocomposite, in comparison with orthoferrite samples, are shown in Fig. 5b. The $M(H)$ curves of the BYFO samples exhibit general similarity and include hysteresis loops, indicating a consistent type of magnetic ordering at RT. Furthermore, the $M(H)$ curves of the nanocomposites approach saturation at approximately 5 kOe, which qualitatively distinguishes them from the $BiFeO_3$ and $YFeO_3$ curves.

It can also be seen that the BYFO-04-09 samples exhibit a significantly higher total magnetization M compared to the pure orthoferrite samples. Among them, the BYFO-05, -06, and -07 samples show the highest magnetic response. These samples with enhanced magnetization contain an excess of iron (see Table S2) and impurities of iron-containing magnetic phases (Fig. 1). Analysis of Mössbauer spectra revealed that the BYFO-05-06-07 samples have a higher proportion of sextets corresponding to the Fe^{3+} states in magnetite Fe_3O_4, which likely enhances the net magnetic response of these composites. In this regard, ferrimagnetism is the most probable type of ordering contributing to the enhanced magnetic response of these materials. It should be noted that the shape of the curves for all doped samples is similar to the $M(H)$ curve of $BiFeO_3$, but with a significantly higher M values (the $M(H)$ curves show a characteristic loop curvature in low fields). This suggests a similar magnetic behavior of the primary phase, while the overall response is dominated by the secondary magnetic inclusions.

In typical $Bi_{1-x}Y_xFeO_3$ solid solutions, the magnetization usually does not exceed 1–2 emu/g, as it originates solely from canted antiferromagnetism. However, the observed $M_s \sim 8$ emu/g are characteristic of ferrimagnetic inclusions, such as Fe_3O_4 clusters. Since M_s correlates with the iron excess rather than with the yttrium molar fraction (x), these materials can be considered as nanocomposites consisting of the original bismuth and yttrium orthoferrites and magnetic iron oxides.

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The physical mechanism underlying this effect in nanocomposites based on bismuth orthoferrite is the partial exchange coupling of magnetic states between $BiFeO_3$ to other magnetic components of the nanocomposite through their interfaces [27,28]. A model describing the interaction between the magnetic moments of $Bi_2Fe_4O_9$ and the local uncompensated moments of the antiferromagnetic $BiFeO_3$, mediated by a spin glass interface, was proposed in [27]. Since the BYFO nanocomposites exhibit a more complex phase composition, the exact nature of their magnetization requires further elucidation.

The hysteresis loop parameters for the BYFO samples are shown in Fig. 6. At room temperature, remarkably high values of saturation magnetization (M_{sat}) and remanent magnetization (M_r) were recorded for the BYFO-05-07 samples. This can be attributed to the iron excess δ and the increased concentration of magnetic phases in these samples (Fig. 1). The $M_{sat}(\delta)$ curve is shown on the inset to Fig. 5.

Given that the Y ion is diamagnetic and possesses no intrinsic magnetic moment, the μ_B/Y value represents the degree of local decompensation within the iron antiferromagnetic sublattice induced by the dopant. M_{sat} is strongly dependent on δ rather than the yttrium concentration alone. To gain deeper insight into the origin of magnetism in BYFO system, the effective magnetic moment per yttrium dopant ion (μ_B/Y) was calculated (see Table 2). The calculation of μ_B/Y showed no systematic trend, confirming that the $Bi^{3+} \rightarrow Y^{3+}$ substitution plays a secondary role in the macroscopic magnetic response compared to parameter δ. The peak observed at x=5 (8.1 emu/g) coincides with the maximum iron excess in this sample, suggesting the formation of uncompensated iron spin clusters that overshadow the intrinsic weak ferromagnetism of the parent phase. The H_c value also increases with increasing iron content in the samples. At $x > 0.5$ and $\delta > 0.5$, the H_c value ceases to depend on the composition of the samples.

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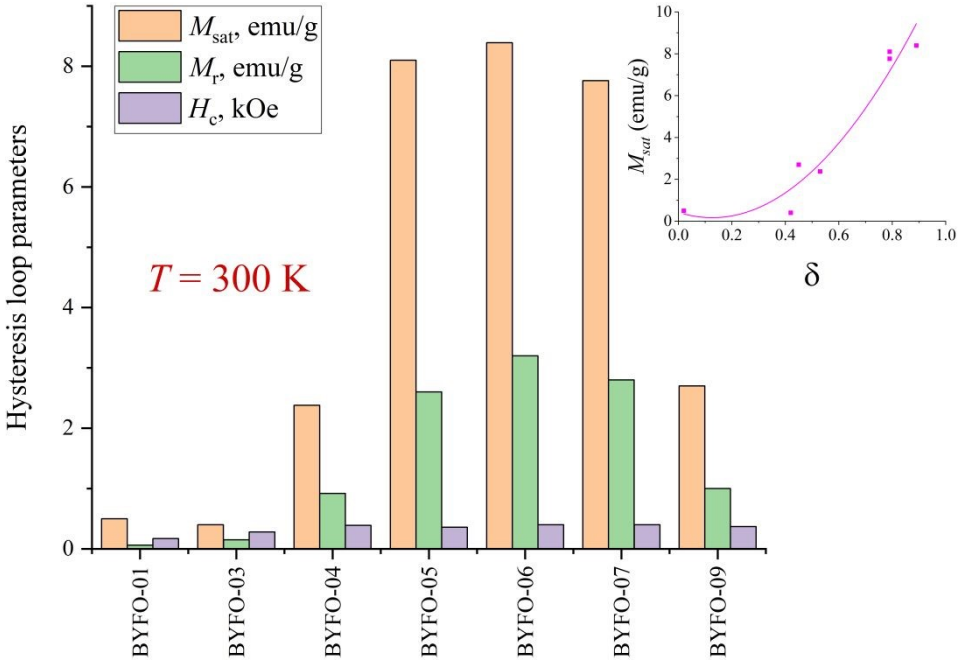

Fig. 5. Remnant magnetization M_r, saturation magnetization M_{sat} and coercivity H_c of the BYFO samples. Inset: M_{sat} measured at 4 kOe as a function of δ

The presence of secondary magnetic phases significantly affects the M_{sat} value, which reaches its maximum in the BYFO-05 and BYFO-06 samples. For these samples, the maximum magnetization was found to be approximately 8 emu/g at RT. As the δ value increases, M_{sat} sharply increases (see insert to Fig. 6). This behavior of M_{sat} value correlates well with the XRD and Mössbauer data discussed above. By tuning the initial composition, we can control the parameter δ and magnetic response of the samples. This provides a pathway to developing new materials with enhanced magnetization, which are potentially useful for nonlinear magnetic media.

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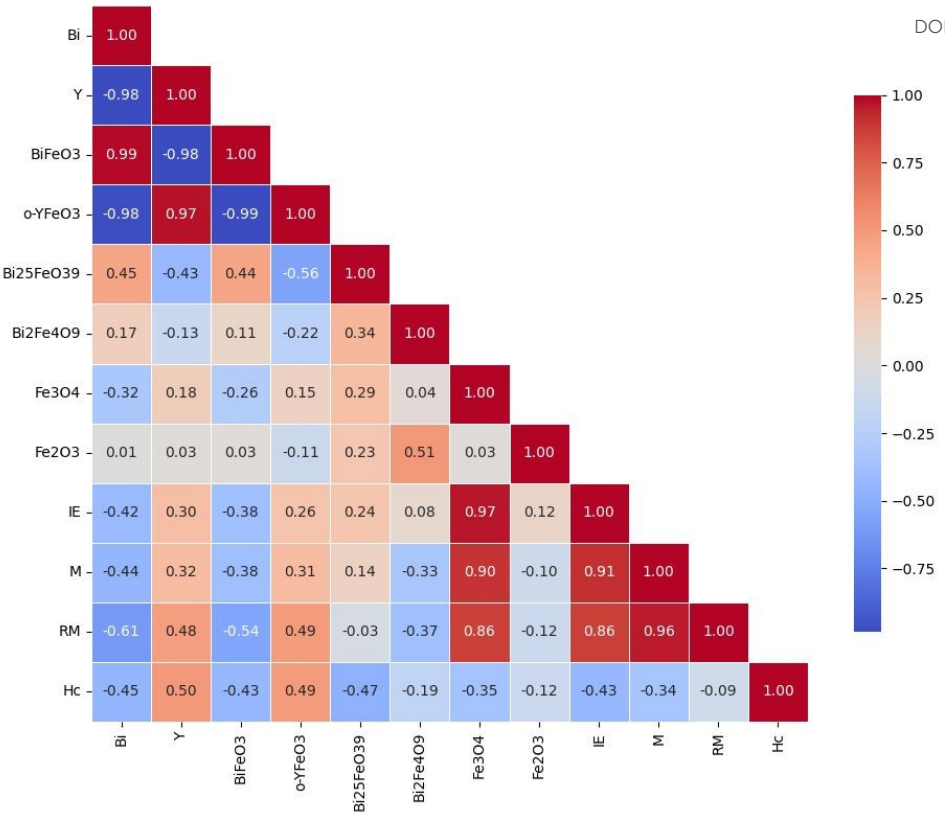

Fig. 6. Pearson's correlation matrix for magnetic properties

To establish the relationships between the composition, phase structure, and magnetic properties of $BiFeO_3$–$YFeO_3$ nanocomposites, a correlation and regression analysis was performed using experimental data and modeling results (Fig. S3). According to the Pearson pair correlation coefficients (PCC), the bismuth content demonstrates a strong positive correlation with the $BiFeO_3$ phase (PCC = 0.99) and a strong negative correlation with *o*-$YFeO_3$ (PCC = –0.98). At the same time, for all measured magnetic parameters - iron excess (Fe_{excess}), magnetization (M), remanent magnetization (M_r), and coercivity (Hc) - the bismuth content correlates negatively (PCC from –0.42 to –0.61). In contrast, the yttrium content has a negative correlation with $BiFeO_3$ (PCC = –0.98) and a positive correlation with *o*-$YFeO_3$ (PCC = 0.97), and also correlates positively with all magnetic properties (PCC from 0.30 to 0.50). This indicates that an increase in the $YFeO_3$ content promotes an increase in magnetization and remanent magnetization, which is consistent with experimental observations: maximum values of M ≈ 8 emu/g at 4 kOe and Mr ≈ 3 emu/g were recorded at x = 0.5–0.7.

Iron-containing secondary phases, which form in composites of intermediate composition, have the greatest influence on the magnetic properties. The strongest correlations are observed for magnetite Fe_3O_4, which is positively correlated with Fe_{excess} (PCC = 0.97), M

(0.90), and Mr (0.86), and only weakly negatively with Hc (–0.35). This confirms that the presence of the ferrimagnetic Fe_3O_4 phase is the main factor enhancing the magnetic response and increasing the total magnetization in the BYFO-05–07 composites. In addition, the *o*-$YFeO_3$ phase shows positive correlations with Mr (0.49) and Hc (0.49), reflecting the contribution of crystalline anisotropy and spin canting to the increase in coercivity. In contrast, $BiFeO_3$ exhibits a negative correlation with Mr (–0.54), and the $Bi_{25}FeO_{39}$ phase with Hc (–0.47), indicating their paramagnetic or weakly ferromagnetic nature, which reduces the overall magnetic response of the composite. Eight models, including polynomial regression and machine learning methods, were used to quantitatively evaluate the relationships between composition and magnetic properties (Fig. S3). The CatBoost and GPR models demonstrated the highest accuracy values ($R^2 = 1.0$) for all parameters, indicating a high adequacy of the nonlinear description of the dependencies. The polynomial model also provided good results for Fe excess ($R^2 = 0.93$), Mr (0.91), and M (0.84), confirming the presence of quadratic components in the composition-dependent properties. The eXGBoost and SVR models showed satisfactory results ($R^2 = 0.50–0.95$), while the linear ElasticNet and eLasso models did not capture the relationship ($R^2 \approx 0$), indicating the complex nonlinear nature of the magnetic behavior of the composite. A combined analysis of correlations and modeling shows that enhanced magnetic properties are observed with increasing yttrium content and the formation of Fe_3O_4 and *o*-$YFeO_3$ phases, while the predominance of $BiFeO_3$ and $Bi_{25}FeO_{39}$ leads to weakening of magnetization and coercivity. The most pronounced magnetic characteristics are achieved at a $BiFeO_3$:$YFeO_3$ ratio of ≈ 1:1 (x ≈ 0.5–0.7), when ferrimagnetic components Fe_3O_4 and Fe_2O_3 form at the phase boundary, facilitating exchange interactions between the spin subsystems. This combination of antiferromagnetic and ferrimagnetic phases ensures the formation of interface-induced ferrimagnetism and explains the nonlinear increase in M and Mr with increasing $YFeO_3$ content.

3.5. Visible-light absorbance and photocatalytic activity

The photocatalytic properties of yttrium-doped bismuth orthoferrites were evaluated by photodegradation of the organic dye RhB in the presence of hydrogen peroxide under visible light. The temporal change in the absorption spectra during the photodegradation of the RhB solution under the influence of the BYFO-04 photocatalyst and H_2O_2 under visible light is shown in Figure 7a. A slight shift in the main absorption peak was also observed with other samples, indicating the conversion of RhB to Rh-110 during N-deethylation indirect photodegradation in the presence of BYFO [55].

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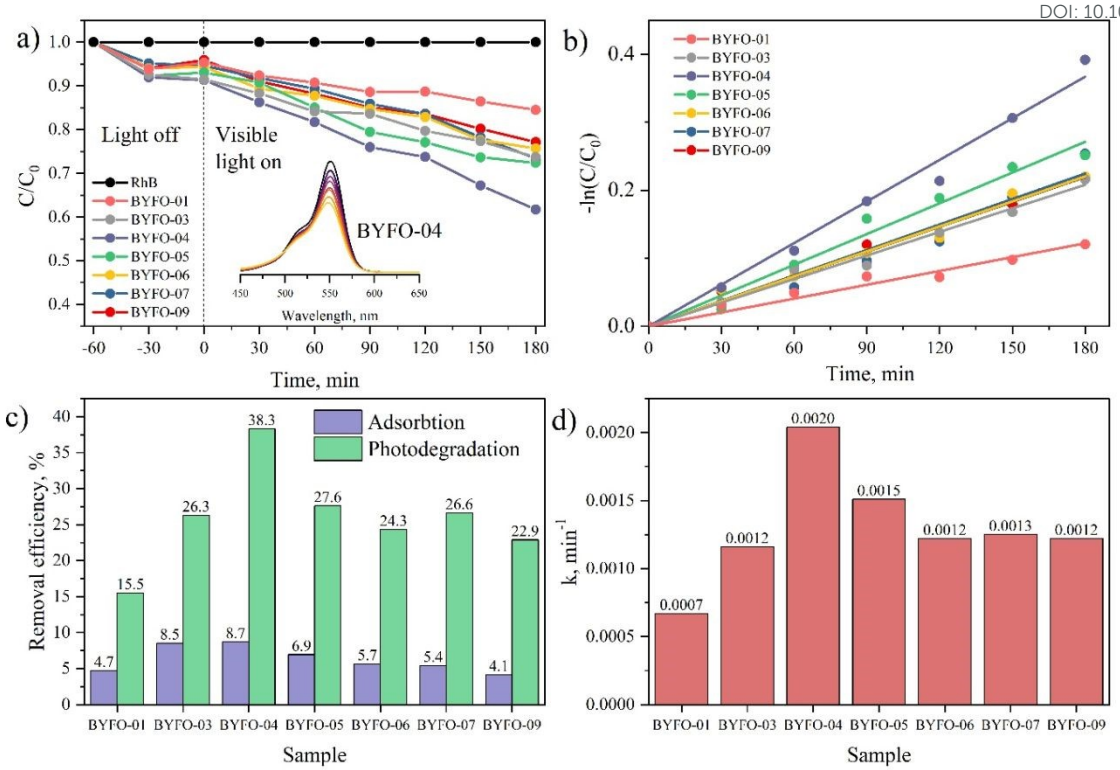

Fig. 7. Photocatalytic performance of BFO-YFO samples under visible light: (a) Fenton-like degradation of dye; absorption spectra of RhB decolorization (Insert a); (b) pseudo-first-order kinetic plots; (c) Removal efficiency during adsorption and photodegradation of RhB; (d) reaction rate constants

Figure 7c shows the time dependence of RhB photodegradation in the presence of BYFO photocatalysts and H_2O_2. When adsorption-desorption equilibrium is reached, the highest degree of adsorption is observed in the BYFO-04 sample, which is the best photocatalyst in the BYFO series. This sample photodegraded about 40% of RhB dye within 180 minutes of irradiation with visible light. According to XRD data, the ratio of the main phases $BiFeO_3$/$YFeO_3$ in this sample is approximately 1:1 (Fig. 2). It can be assumed that such a phase ratio ensures the creation of a stable interface between the components of the BYFO nanocomposite, which leads to an increase in its photocatalytic efficiency. A further increase in concentrations leads to a decrease in photocatalytic activity and the formation of a plateau in degradation efficiency (Fig. 7b).

One of the possible reasons for the highest photoactivity of the BYFO-04 sample with the addition of hydrogen peroxide to the reaction may be the presence of a photocatalytically active semiconductor Fe_2O_3 in this sample (see Figure 2). Compared to other samples, the formation of the impurity compound Fe_2O_3 can serve to suppress electron-hole recombination

by the effect of capturing electrons from the conduction band by Fe^{3+} ions and further accelerate the Fenton-like reaction by initiating the activation of H_2O_2 with the formation of active ·OH radicals [34], which can be explained by the redox reaction of Fe^{+2}/Fe^{+3} ions illustrated by the equations:

$$H_2O + h^+ \rightarrow \cdot OH$$

$$Fe^{+3} + e^- \rightarrow Fe^{+2}$$

$$Fe^{+2} + H_2O_2 \rightarrow Fe^{+3} + OH^- + \cdot OH$$

In Figure 10d, it can be seen that a fairly good linear correlation was found ($R > 0.98$), indicating that the reaction is a pseudo-first order reaction. To quantitatively understand the kinetics of the RhB degradation reaction, a pseudo-first order model expressed by Equation (2), which is typically used at low initial contaminant concentrations, was applied [56]. As stated above, the presence of impurity iron oxides in BYFO-04 leads to an increase in the photodegradation rate constant to k=2.04·10^{-3} min^{-1}.

$$\ln\left(\frac{C_0}{C}\right) = kt \quad (2)$$

where C_0 is the initial concentration of the dye, C is the concentration of the dye after t minutes, k is the reaction rate constant (min^{-1}), t is time.

To determine the compositional dependence of the photocatalytic activity of $BiFeO_3$–$YFeO_3$ nanocomposites, a correlation and regression analysis was performed using experimental parameters (k, RE, $C_{reacted}$, C_{final}, $t_{10\%}$, Ads) (Fig. 8). According to the calculations of the Pearson pair correlation coefficients (PCC), the bismuth content has a strong positive correlation with the $BiFeO_3$ phase (PCC = 0.98) and a strong negative correlation with the *o*-$YFeO_3$ phase (PCC = –0.97). For photocatalytic parameters, a negative correlation is observed between the bismuth content and the rate constant (k, –0.44), the decomposition efficiency (RE, –0.34), and the amount of reacted substance ($C_{reacted}$, –0.34), while a positive correlation is observed with the final dye concentration (C_{final}, 0.34), the time to reach 10% degradation ($t_{10\%}$, 0.29), and the degree of adsorption (Ads, 0.19). Thus, an increase in the $BiFeO_3$ proportion is accompanied by a decrease in photocatalytic activity. In contrast, increasing the yttrium content, which correlates negatively with $BiFeO_3$ (PCC = –0.97) and positively with *o*-$YFeO_3$ (PCC = 0.96), has the opposite effect: for the k, RE, and Creacted parameters, the correlation is positive (0.39–0.50), while for Ads, $t_{10\%}$, and C_{final}, it is negative (–0.14 to –0.39). This indicates that increasing the proportion of $YFeO_3$ and associated oxide phases accelerates photodegradation and increases the efficiency of organic compound decomposition under the influence of visible light.

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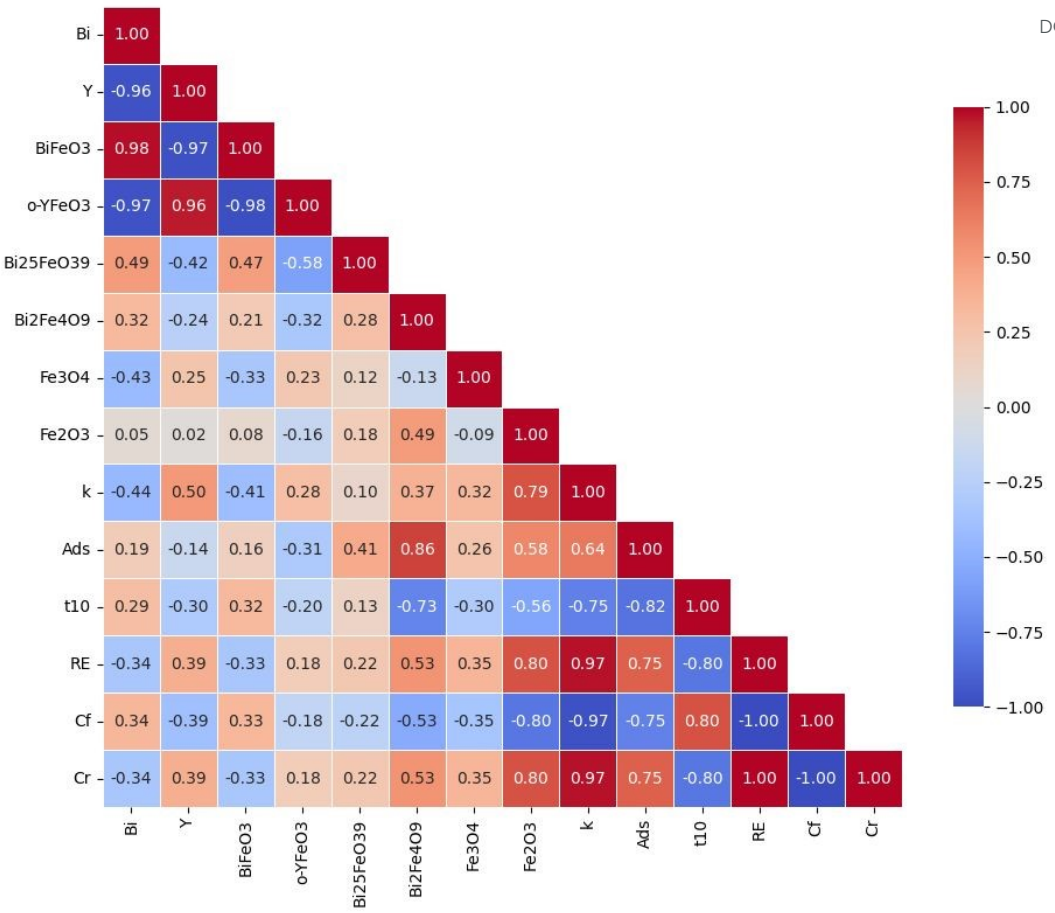

Fig. 8 Pearson's correlation matrix for photocatalytic properties

The contribution of secondary phases should be noted separately. The appearance of the $Bi_2Fe_4O_9$ phase is accompanied by a strong positive correlation with Ads (0.86), RE (0.53), $C_{reacted}$ (0.53) and a negative one with t10% (–0.73), C_{final} (–0.53), indicating an increase in photo-Fenton-like activity with increasing its content. Similarly, the Fe_2O_3 phase exhibits a positive correlation with Ads (0.58), RE (0.80), $C_{reacted}$ (0.80) and a negative one with $t_{10\%}$ (–0.56), C_{final} (–0.80), confirming its role in accelerating dye degradation processes due to the generation of active ·OH radicals.

Eight models, including polynomial regression and machine learning algorithms, were used to quantitatively describe the dependencies (Fig. S4). The best results across all parameters were achieved for the CatBoost (R^2 = 1.0) and GPR (R^2 = 0.94–1.0) models, confirming the presence of pronounced nonlinear relationships between composition and photocatalytic performance. The polynomial model demonstrated high R^2 values for Ads (0.996), k (0.93), $t_{10\%}$ (0.96), and RE (0.89), indicating an adequate description of the effect through a quadratic relationship. The eXGBoost and SVR algorithms showed moderate results (R^2 = 0.45–0.76), while the ElasticNet and eLasso linear models were unable to capture the

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patterns ($R^2 \approx 0$), confirming the complex nonlinear nature of photodegradation processes in heterogeneous systems. Thus, the simulation confirmed that increasing the yttrium content and the formation of the $YFeO_3$ phase, as well as the presence of photocatalytically active Fe_2O_3 and $Bi_2Fe_4O_9$ phases, contribute to an increase in the degradation rate of organic dyes and an increase in the efficiency of photocatalysis. The highest values of the rate constant (k = 2.04 10^{-3} min^{-1}) and degradation efficiency (RE) are observed in samples with a close ratio of $BiFeO_3$:$YFeO_3$ ≈ 1:1 (x ≈ 0.4–0.5), which is consistent with the experimental results and confirms the role of interfacial interactions in enhancing photo-Fenton-like processes.

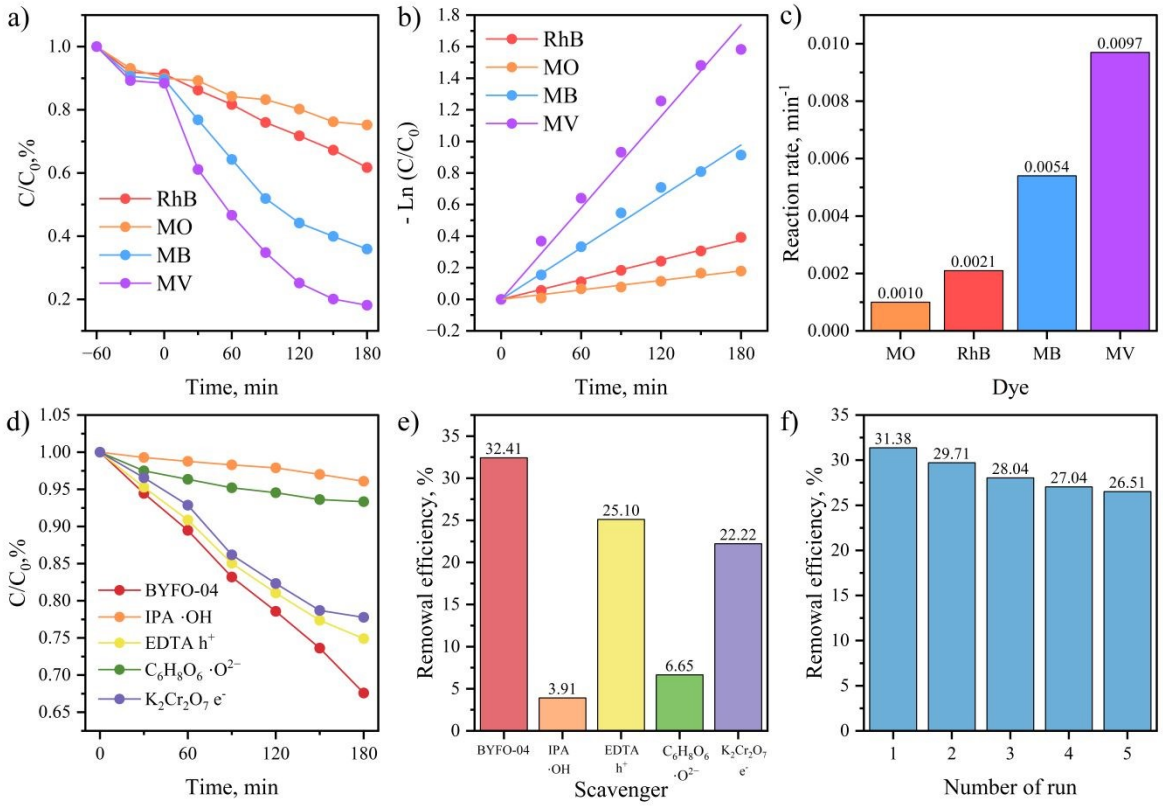

Fig. 9 Photocatalytic performance of BYFO-04 sample under visible light: (a) Fenton-like degradation of dyes; (b) pseudo-first-order kinetic plots; (c) reaction rate constants; (d-e) scavenger tests for RhG photodegradation; (f) cyclic stability test.

To assess the practical applicability of the synthesized BYFO-04 photocatalyst, its photocatalytic activity was evaluated using a range of model organic dyes (Fig. 9 a-c). The material demonstrated high degradation efficiency toward thiazine and triarylmethane dyes, achieving 65% and 80% decomposition of methylene blue and methyl violet, respectively. In contrast, the degradation efficiency for azo dyes was limited to approximately 25%, which can

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be attributed to their greater structural stability under the applied conditions. Reaction rate constants are presented in Fig. 9 d.

To elucidate the mechanism of the Fenton-like degradation of rhodamine B catalyzed by the synthesized material, a series of radical scavenging experiments were conducted (Fig. 9 d-e). Ascorbic acid was employed to scavenge superoxide radicals ($\cdot O_2^-$), isopropyl alcohol (IPA) was used to trap hydroxyl radicals (·OH), disodium ethylenediaminetetraacetate (EDTA) served as a hole (h^+) scavenger, and potassium dichromate ($K_2Cr_2O_7$) was utilized to capture photogenerated electrons (e^-). The results indicate that hydroxyl radicals (·OH) and superoxide radicals ($\cdot O_2^-$) play dominant roles in the degradation process, whereas photogenerated holes (h^+) and electrons (e^-) contribute to a lesser extent [57]. Moreover, cyclic stability tests revealed a gradual decline in catalytic activity, with an approximate 5% loss in efficiency observed by the fifth cycle (Fig. 9 f).

The diffuse reflectance spectra of the samples presented in Fig. 10a. All the samples demonstrate low reflectance at 400-550 nm that indicates the effective absorbance of visible light spectrum. The bends of curves demonstrate the strong interaction with visible light in its full diapason for all samples. It can be noticed that the reflection rate systematically decreases with increase of yttrium content, except YFO sample. The band gap energy values were evaluated by extrapolation of the linear Tauc plots parts (Fig. 10b) and summarized in Table S4. The band gap energy values (Fig. 9c) for the BFO and YFO samples were 2.0 and 1.94 eV, respectively, which is close to previous reports (4, 14). The E_g values of $BiFeO_3$ slightly increase with increase of yttrium content up to 2.04 eV, within the error of the determination method. For the *o*-$YFeO_3$ phase (BYFO-0.3-0.5, 1) there is the same behavior with insignificant change of band gap energy. However, for the several samples (BYFO-0.6-0.9) the noticeable decrease of E_g is observed, which can be associated with their solid-solution nature.

The band gap structure parameters were evaluated by followed empirical formulas: $E_{VB} = \chi - E_e + 0.5 \cdot E_g$ and $E_{CB} = E_{VB} - E_g$, where $E_e = 4.5$; χ is electronegativity χ (YFO) = 5.60 eV, χ (BFO) = 5.89 eV. The calculated values of the conduction band edge potential (E_{CB}) and valence band edge potentials (E_{VB}) are presented in Table S4 and the band gap structure of the Y04 is visualized in Fig.10.

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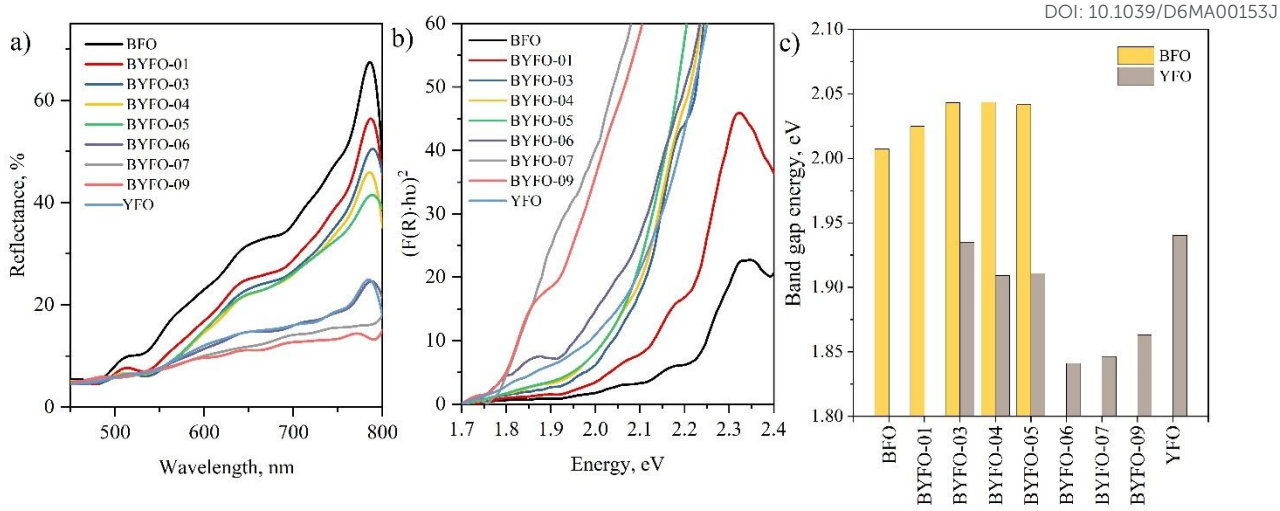

Fig. 10. Diffuse reflectance spectra (a), Tauc plots (b), and band gap energy values (c) of the BFO-YFO samples.

The activity of both nanocomposite materials under visible light irradiation (E_g (BFO) = 2.04 eV, E_g (YFO) = 1.91 eV) was previously mentioned.

$$BiFeO_3 + h\nu \rightarrow e^-_{CB} + h^+_{VB}$$

$$YFeO_3 + h\nu \rightarrow e^-_{CB} + h^+_{VB}$$

The calculated CB and VB values were 0.37 eV and 2.41 eV for BFO, and 0.16 eV and 2.05 eV for YFO providing the heterojunction formation. The possibility electron transfer from $YFeO_3$ CB to $BiFeO_3$ CB and hole transfer from $BiFeO_3$ VB to $YFeO_3$ VB prevents the coupling of electron-hole pairs and increases photocatalytic activity (Figure 11). The wide band gaps and high VB values of both nanocomposite components make feasible several ·OH radical generation reactions.

$$OH^- + h^+ \rightarrow \cdot OH$$

$$H_2O + h^+ \rightarrow \cdot OH + H^+$$

$$H_2O_2 + e^- \rightarrow \cdot OH + OH^-$$

$$O_2 + e^- \rightarrow \cdot O^{2-}$$

$$\cdot O_2^- + H_2O \rightarrow \cdot OH_2 + OH^-$$

$$\cdot OH_2 + \cdot OH_2 \rightarrow O_2 + H_2O_2$$

$$H_2O_2 + \cdot O^{2-} \rightarrow \cdot OH + O_2 + OH^-$$

The ·OH radicals are known to be very effective in organic pollutants degradation.

$$RhB + \cdot OH \rightarrow CO_2 + N_2 + H_2O$$

The presence of Fe^{3+} ions in both nanocomposite materials leads to multiple ·OH radicals' generation in Fenton-like process.

$$Fe^{3+} + e^- \rightarrow Fe^{2+}$$

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + \cdot O_2^- + H_2O$$

That explains the high photocatalytic activity of the $BiFeO_3$–$YFeO_3$ heterojunction nanocomposite in the process of Fenton-like photodegradation of RhB. The reduced electron-hole pairs coupling increased ·OH radicals' generation, and high level of visible light absorption make this photocatalyst a promising material for advanced oxidation processes of organic pollutants decomposition.

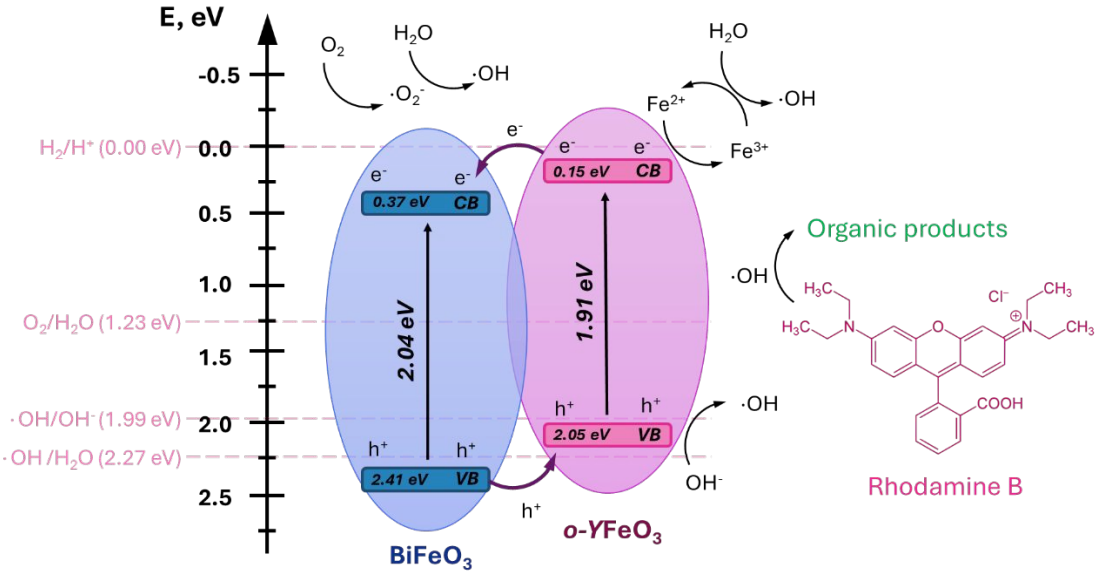

Fig. 11. Mechanism of photo-Fenton degradation of RhB over BFO-YFO nanocomposites

An increase in the photocatalytic activity of $BiFeO_3$-$YFeO_3$ nanocomposites can be realized by varying the iron content in the target product, which can be influenced by synthesis conditions, such as the type of organic fuel and the fuel/oxidizer ratio in the initial composition. Properties such as magnetically ordered state at room temperature and obvious photocatalytic activity of the synthesized materials are promising for efficient separation of the photocatalyst from the reaction medium.

4. Conclusion

Nanocrystalline materials based on the $x$$BiFeO_3$-(1-$x$)$YFeO_3$ system were synthesized using the glycine-nitrate combustion method. The obtained materials have an average crystallite size in the range of 25–90 nm. XRD and EDX data demonstrated the effect of the

initial composition on the phase composition of the target product. At nominal values of x = 0.3-0.7, nanocomposites samples were formed. It was found that BYFO-04 nanocomposite with a ratio approximately 1:1 between bismuth and yttrium orthoferrites, exhibits increased photocatalytic activity compared to the other samples.

The magnetic properties of the obtained materials were investigated by Mössbauer spectroscopy and magnetometry. At RT, all synthesized materials exhibit magnetic order. The results showed that the BYFO materials have improved magnetic properties, demonstrating in an increase in total magnetization by several times compared to pure orthoferrites. The BYFO-0.5 and BYFO-0.7 nanocomposites showed an improved magnetic response, with specific and saturation magnetization values of approximately 8 emu/g at a field of 4 kOe. The discovered properties of the synthesized materials are promising for the effective separation of photocatalysts from the reaction medium.

Author contributions

S. M. Tikhanova – Formal analysis, Investigation, Visualization, Writing – original draft, Writing – review & editing; **M.P. Volkov** – Data curation, Formal analysis, Investigation; **I.V. Buryanenko** – Data curation, Formal analysis, Investigation; **V.G. Semenov** – Data curation, Formal analysis, Investigation; **Zh. Orazovd** –Data curation, Formal analysis, Investigation; **V.I. Romanovski** - Data curation, Formal analysis, Investigation, Visualization, Writing – original draft, Writing – review & editing; **V.I. Popkov** – Project administration, Supervision, Validation, Writing – review & editing; **N.A. Lomanova** - Conceptualization, Methodology, Project administration, Supervision, Validation, Writing – review & editing.

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Conflict of interest

The authors declare no competing interests.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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Data Availability Statement

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- No primary research results, software or code have been included and no new data were generated or analysed as part of this review.