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Advances made by Belgrade's group in research of oscillatory reactions

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Abstract

Oscillatory dynamic states as one form of selforganization of nonlinear systems can be found in almost all sciences, like mechanics, physical chemistry or biomedicine. Although origin of these oscillations is different, computational challenges in modelling oscillatory phenomena remain similar in all fields. Since 1979 researchers from Belgrade's group perform systematic examinations of oscillatory reactions. As stability of steady states is the central point in modelling oscillatory reactions, in last 10 years they have adapted and improved powerful tool of the Stoichiometric Network Analysis for this goal. Moreover, bifurcations of few types were identified in several models of oscillatory reactions. Even very complex chaotic motions in phase space were characterized and quantified by several numerical techniques. Multiple time scale behaviour is found within the core of the complex dynamic behaviour of mixed-mode oscillations. Analytical applications were developed, too.

Keywords: Oscillatory reactions, Nonlinear dynamics, Stoichiometric Network Analysis, Numerical techniques for detection of periodic and aperiodic dynamic states, Analytical determination

1. Introduction

Some nonlinear reaction systems, when they are far from thermodynamic equilibrium, can be in an oscillatory dynamic state. These oscillations can be detected and monitored in time through the concentrations, temperature, electrochemical, spectroscopic or any other measurable properties of investigated reaction system. Such oscillatory reaction systems in which spontaneous periodic changes in the concentration of intermediates are observed were discovered accidentally. The detailed description of oscillatory reactions discovery can be found in several papers (Winfree 1984; Zhabotinskii 1991; Stávek et al. 2002; Epstein et al. 2006; Field et al. 1985; Kolar-Anić et al. 2017a; Kolar-Anić et al. 2017b; Kolar-Anić et al. 2017c; Kolar-Anić et al. 2017d), and books related to this subject (Nicolis et al. 1977; Epstein et al. 1998; Field et al. 1985). Investigations of such systems essentially changed our understanding of reaction systems in general and the way that contemporary scientists were thinking about them. It was found that these phenomena can be explained only by the main relations of

nonlinear dynamics and basic statements of Nonequilibrium thermodynamics, two compatible sciences that investigate general phenomena of self-organization.

1.1. Discovery of homogenous oscillatory reactions - The short overview

The first study of experimentally controlled oscillatory dynamic states in a homogeneous reaction system was published in 1921 (Bray 1921). In this paper William C. Bray investigated the dual role of hydrogen peroxide during its decomposition in an acid solution of iodate ions.

$$2\,H_2O_2 \xrightarrow[IO_3^-,\ H^+]{k_D} 2H_2O + O_2 \qquad (D)$$

Since in the considered solution the global reaction (D) is the result of presence of two parallel processes where the reduction of iodate to iodine (R) and the oxidation of iodine to iodate (O) are performed,

$$2IO_3^- + 2H^+ + 5H_2O_2 \xrightarrow{k_R} I_2 + 6H_2O + 5O_2 \qquad (R)$$

$$I_2 + 5H_2O_2 \xrightarrow{k_O} 2IO_3^- + 2H^+ + 4H_2O, \qquad (O)$$

he concluded that the periodic domination of one of them can cause the oscillations of intermediate iodine species concentrations. Bray continued these investigations with his student Herman Liebhafsky (Bray et al. 1931; Liebhafsky 1931a; Liebhafsky 1931b; Liebhafsky 1932a; Liebhafsky 1932b; Liebhafsky et al. 1933; Liebhafsky 1934) and the aforementioned reaction was subsequently named the Bray-Liebhafsky (BL) oscillatory reaction.

Around 1950, Boris Pavlovich Belousov discovered independently the second homogeneous oscillatory reaction. (Field et al. 1985) He observed similar dynamic behaviour in a solution of citric acid, bromate and ceric ions (Ce^{4+}), where the yellow solution periodically cleared and became yellow again. However, he had serious troubles to publish these results. His attempt to publish them in 1951 did not succeed, since general opinion at that time was that this kind of dynamic behaviour is not in accordance with the second law of thermodynamics. After several years, in 1958, he published his work in the Book of abstracts at the Conference of radiation biology. (Belousov 1958) Later, posthumously, his original manuscript from 1951 was published in Russian (Belousov 1981) and in English (Belousov 1985). Anatol M. Zhabotinskii, a graduate student in biophysics, continued Belousov´s initial work during the 1960´s. He replaced the citric acid by malonic acid (Zhabotinskii 1964) and created the chemical system in which various concentration oscillations have been observed. Moreover, he added the redox indicator ferroin, to enable visualisation of the oscillation through periodical colour changes from red to blue and gave rise to spatio-temporal waves in a thin layer. This reaction, named the Belousov-Zhabotinskii (BZ) oscillatory reaction, became a very popular model system and attracted the attention of many scientists working in the fields of Nonequilibrium thermodynamics and Nonlinear dynamics.

The first explanation showing that this kind of dynamic behaviour is possible in homogeneous isothermal reaction systems came from Ilya Prigogine[1] who introduced the concept of nonequilibrium thermodynamics and clearly distinguished entropy production in closed and open systems. Thus, it was demonstrated that nonequilibrium stationary states are not equal to the equilibrium ones, and that selforganization phenomena under these conditions differ from one another. Finally, dissipative structures are introduced to distinguish

[1] Prof. Ilya Prigogine was the Nobel Prize winner for chemistry in 1977.

nonequilibrium selforganization phenomena from the ones found in an equilibrium (Prigogine 1977; Nicolis et al. 1989; Nicolis 1995).

The basic theoretical foundations for modelling the homogeneous oscillatory processes originate from Alfred Lotka who already at that time (1910-1925) postulated the mathematical model appropriate to simulate oscillatory evolution in reaction and population systems (Lotka 1910; Lotka 1920a; Lotka 1920b; Lotka 1925). His investigations together with those performed by Vito Voltera (1926) to explain ecological problems, comprise the Lotka-Voltera model which is the preferred model for describing predator-pray interactions. This model also formed the basis for several famous models, such as Brussellator, (Lefever et al. 1988) Oregonator (Field et al. 1974) and autocatalator, (Gray et al. 1986; Scott 1987; Gray et al. 1990) which were proposed to explain self-organized phenomena obtained experimentally in several complex nonlinear reaction systems (Belousov 1958; Zhabotinskii 1964; Briggs et al. 1973). Although these models had an important role in explaining the existence of stable and unstable nonequilibrium stationary states in nonlinear reaction systems, as well as the possible self-organizing phenomena in these states, a straightforward correlation between reaction species and mathematical variables was not established for a long time. The main problem was in the fact that direct autocatalytic reactions, necessary in the model to simulate oscillatory dynamics, do not appear in real reaction systems. The first model where a direct autocatalytic step was successfully substituted by a realistic autocatalytic loop was proposed in 1987 by Guy Schmitz who was working on the modelling of the Bray-Liebhafsky oscillatory reaction (Schmitz 1987). Building on the original model proposed by Schmitz, a more powerful variant of the model was developed (Kolar-Anić et al. 1992; Kolar-Anić et al. 1995a) that could simulate complex dynamical structures such as mixed-mode oscillations, period doubling and deterministic chaos (Kolar-Anić et al. 2004a; Kolar-Anić et al. 2004b; Ivanović et al. 2008; Ivanović et al. 2009; Kolar-Anić et al. 2010; Ivanović et al. 2011; Čupić et al. 2013). Thus, the connection between experimental and theoretical investigations was established and proved by numerical investigations.

1.2. The investigations of Belgrade's group - historical background

Since 1979 the Belgrade's group emerged with systematic examinations of oscillatory reactions. The first of our results were related to the phenomenological studies of their dynamic behaviours and reaction kinetics (Anić et al. 1985; Anić et al. 1986a; Anić et al. 1986b; Anić et al. 1987; Anić et al. 1988; Anić et al. 1989a; Anić et al. 1989b; Anić et al. 1991; Anić et al. 1996; Anić. 1997a; Anić et al. 1997b; Anić et al. 1998; Anić et al. 2007; Anić et al. 2009; Blagojević 2000; Blagojević et al. 2008; Blagojević et al. 2009; Ćirić et al. 2000; Milenković et al. 2012; Radenković 1997; Stanisavljev et al. 1995; Stanisavljev 1997; Stanisavljev et al. 1998a; Stanisavljev et al. 1998b; Stanisavljev et al. 2002; Stanisavljev et al. 2011; Vukojević et al. 2000; Vukojević et al. 2002). Furthermore, various methods were developed for formal kinetics of homogenous oscillatory process (Anić et al. 1986a; Anić et al. 1987; Anić et al. 1988; Anić et al. 1996; Anić et al. 2007), as well as for the stability analysis of the postulated models (Kolar-Anić et al. 1995b; Schmitz et al. 2000). All investigations were used for different purposes, starting with the development of new analytical methods (Pejić et al. 2001; Pejić et al. 2003; Pejić et al. 2005; Pejić et al. 2006; Pejić et al. 2007a; Pejić et al. 2007b; Pejić et al. 2007c; Pejić et al. 2009; Pejić et al. 2011; Pejić et al. 2012; Pejić et al. 2013; Pejić et al. 2014a; Pejić et al. 2014b; Pejić et al. 2016; Vukojević et al. 1999; Vukojević et al. 2001) and the methods for determining the catalyst's activity (Čupić et al. 1995; Terlecki-Baričević et al. 1995; Pejić et al. 2001; Anić et al. 2009; Maksimović et al. 2011), as well as the influence of external fields on the evolution of the chemical oscillator (Stanisavljev et al. 2004; Stanisavljev et al. 2005; Stanisavljev et al. 2006; Stanisavljev et al. 2014) to modelling of biochemical processes (Jelić et al. 2008; Jelić et al. 2009; Čupić et al. 2016b; Čupić et al. in press; Kolar-Anić 2017d;

Marković at. al 2011a; Čupić et al. in press; Marković et al. 2011b; Marković et al. 2016;) etc. (Anić et al. 1994; Čupić et al. 1996; Begović et al.2004).

Main contributions of the Belgrade group to the research of oscillatory reactions with emphasis on the results from the latest 10 years are given in the Section 2. The Belgrade group gave its contribution to both experimental investigation and theoretical description of several oscillatory systems including BL, BZ, Briggs-Rauscher (BR), hypothalamic–pituitary–adrenal (HPA), and others. In Subsection 2.1 the experimental investigations related to controlled generation of various dynamical states and bifurcations between them including precise quantification of their complexity using several methods, such as Power spectrum, Lyapunov exponents, Return maps, etc, are presented. Next, also important track of experimental investigation is given in Subsection 2.2. In fact, this part refers to the formal kinetic analysis of homogeneous oscillatory reactions using (the traditional way and) the methods specially developed for oscillatory reactions. Finally, the developed applications of oscillatory reaction systems in analytical procedures for determination of pharmaceutically active substances and their antioxidative activity are given in the Subsection 2.3. On the other side, the results of modelling the considered reaction systems are presented in the Subsection 2.4. For achieving best performances of the developed models, sophisticated mathematical apparatus of SNA was used and improved. Also, there is a brief review of the influences of microwave (MW) and radiofrequency (RF) fields on the BL reaction (Subsection 2.5). Instead of conclusions, our vision of future trends in the field is given in the Section 3.

2. Contemporary research in Belgrade group

From the beginning as well as in the last ten years, we analyze oscillatory dynamics of reaction systems, more precisely, dynamical states of chemical, physicochemical and biochemical reaction systems, both experimentally and theoretically using numerical method to correlate obtained results. Although our systems are different from mechanical oscillators, we also examine stability of the states, oscillatory regions, bifurcation points, periodic and aperiodic oscillations (deterministic chaos), the standard notions in Nonlinear dynamics. Necessary mathematical equipment is similar or even the same as the one applied in other scientific fields. Thus, the obtained deterministic chaos in reaction system must be analysed and proved by common methods for deterministic chaos.

2.1 Controlled generation of various dynamic states

Controlled generation of various dynamic states, from regular oscillatory to the chaotic ones, has been our main aim from the beginning. The experimental results stimulated theoretical investigations and *vice versa* (Fig.1). Thus, experimentally obtained mixed-mode and chaotic states influenced their modelling. When we found that our model of the BL reaction confirms the existence of the deterministic chaos in this system (Kolar-Anić et al. 2004a; Kolar-Anić et al. 2004b) the attention in our experimental investigation moved to even more complex oscillations (Schmitz et al. 2006). Consequently, the exotic dynamic states were generated under controlled conditions in the BL system (Pejić et al. 2011; Bubanja et al. 2016) and also in the BR reaction perturbed by phenol (Čupić et al. 2014). As a result, the possibility to generate and control the intermittent dynamics in the BL reaction (Fig. 2) is demonstrated for the first time, without addition of any perturbing substances.

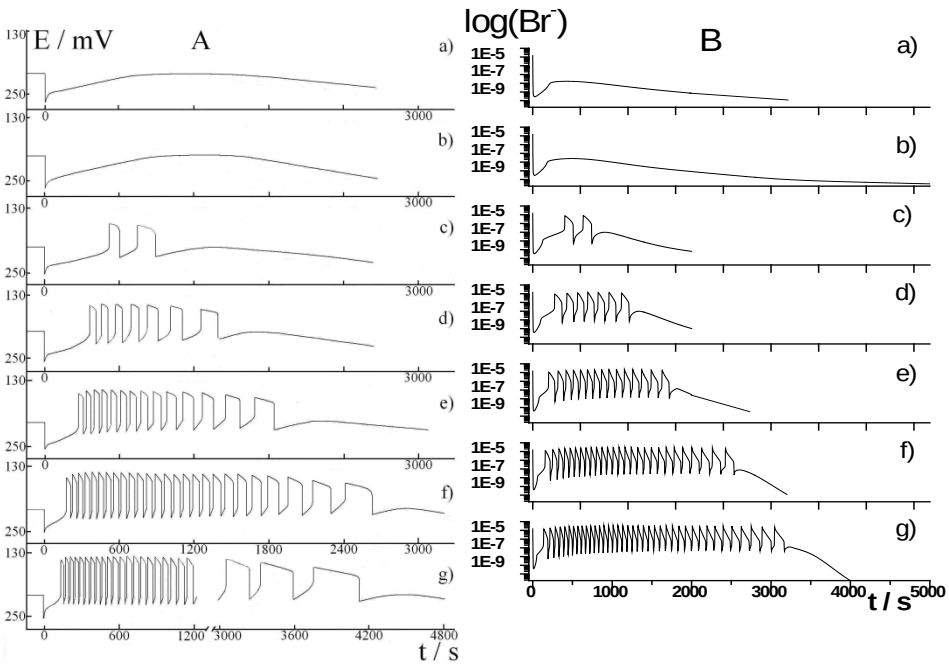


Fig. 1. Oscillograms in BZ system obtained experimentally A) and numerical simulation B) for the temperature 35°C and initial concentration of malonic acid (mol dm^{-3}): a) $8{,}00 \times 10^{-3}$; b) $9{,}00 \times 10^{-3}$; c) $1{,}20 \times 10^{-2}$; d) $1{,}60 \times 10^{-2}$; e) $2{,}20 \times 10^{-2}$; f) $3{,}20 \times 10^{-2}$; g) $4{,}30 \times 10^{-2}$; Oscillograms obtained by numerical simulation are based on BG (1-12) model

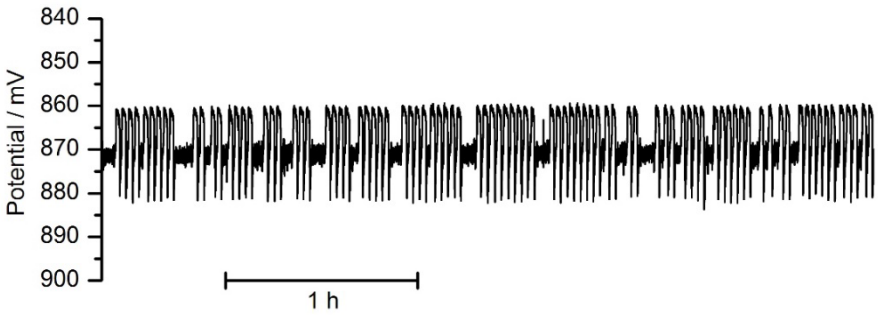
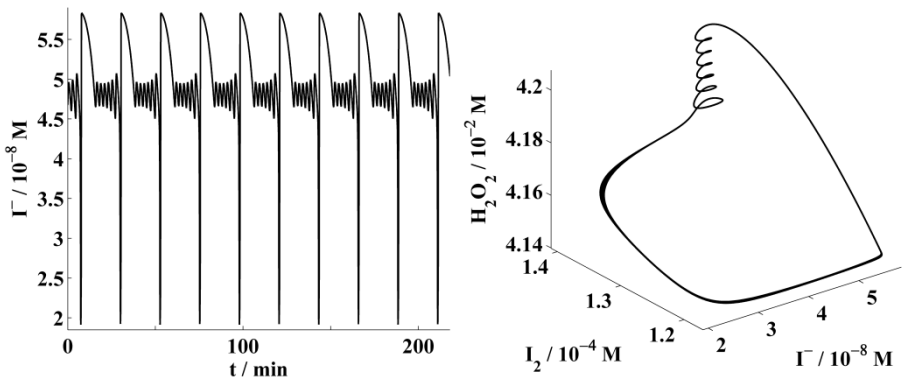


Fig. 2. Oscillogram (potential *vs* time) of the intermittent dynamic state obtained in Bray-Liebhafsky oscillatory reaction generated in continuously fed stirred tank reactor (CSTR)

Methods for characterization of intermittencies were developed and deterministic nature of the phenomena is proved in both BL and BR system. In general, exotic dynamic states (Fig.3) were characterized by various mathematical techniques, starting from the Lyapunov exponents, Poincare sections, return maps, multifractal distributions, *etc* (Kolar-Anić al. 2006; Pejić et al. 2009; Čupić et al. 2014; Ivanovo et al. 2008; Ivanović et al. 2009; Ivanović et al. 2011; Čupić et al 2013; Blagojević et al. 2015; Bubanja et al. 2016; Čupić et al. 2016a).



(a) (b)

Fig. 3. Numerical simulations of the complex mixed mode oscillations in the BL reaction model. (a) Time series; (b) Phase space attractor

The dynamic states and kinetics of Belousov–Zhabotinsky reaction was also examined (Blagojević et al. 2008; Blagojević et al. 2009). We have examined the influence of temperature and malonic acid concentration on the dynamic states of the BZ system. Improved variant of the model was proposed and excellent agreement is achieved between the experiment and numerical simulations (Blagojević et al. 2011; Blagojević et al. 2013).

2.2 Evaluation of kinetic parameters

Almost all methods for evaluation of kinetic parameters of oscillatory reactions are developed on the BL reaction (Anić et al. 1986a; Anić 1987; Anić et al. 1988; Anić et al. 1989a; Anić et al. 1996a; Anić et al. 1996b; Anić 1997a; Anić et al. 1997b) and later applied to other ones (Blagojević et al. 2000; Blagojević et al. 2008; Blagojević et al. 2009). So, it was the case with the rate constants of overall process (D), as well as the reaction routes (O) and (R) and its corresponding apparent activation energies and also order and pseudo orders of overall reaction (D). The two competing routes (R) and (O) were clearly obtained both experimentally and theoretically by application of the same phenomenological methods on the experimentally obtained results as well as on the proposed models.

The BL reaction was also used as the reaction medium for the catalyst characterization (Anić et al. 2009; Maksimović et al. 2011). Cobalt ions on porous polymer support were used to perturb the kinetics of the BL system. Catalytic activity was then evaluated based on apparent activation energies. By calculating apparent activation energies of the reduction and oxidation processes in the BL reaction and their dependence on the mass of added catalyst, the oxidative catalytic activity of the tested catalyst activity was evaluated.

2.3 Analytical procedures

The oscillatory reactions with at least two reaction routes and very low concentrations of intermediate species are extremely sensitive on perturbations and therefore appropriate for analytical purposes. Using them, analyte pulse perturbation techniques (Fig. 4) were developed for determination of paracetamol, ascorbic acide, morphine, 6-monoacetylmorphyne, piroxicam, and also for uric acid in human urine (Pejić et al. 2006; Pejić et al. 2007a; Pejić et al. 2007b; Pejić et al. 2007c; Pejić et al. 2012; Pejić et al. 2014).

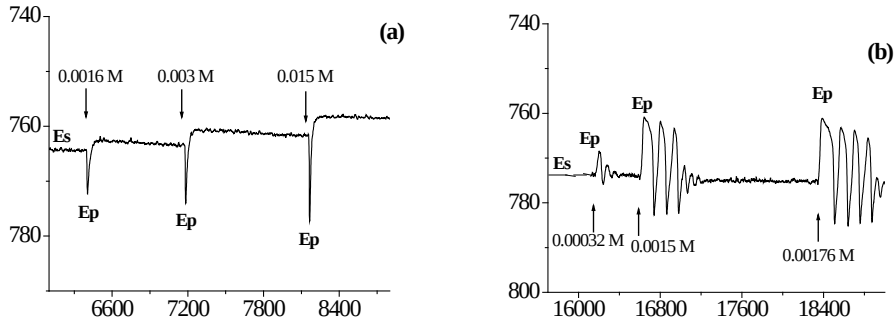


Fig. 4. Potentiometric responses of BL analyte matrix, which is originally in the stationary state, obtained after the pulse perturbation by various concentrations of vitamin B1 (a) and B2 (b). Arrows indicate the moment of perturbation

2.4 Modeling

For controlled generation of various dynamic states and prediction of dynamic states of the reaction system, the model of the process ought to be postulated. Modeling procedure is a serious task. It depends very much on the system under consideration. For this purpose, theory of the Stoichiometric Network Analysis was improved and used for identification of the instability regions in several models of the oscillatory reactions (Jelić et al. 2008; Schmitz et al. 2008; Jelić et al. 2009; Kolar-Anić et al. 2010; Čupić et al. 2011; Marković et al. 2011a; Maćešić et al. 2012; Maćešić et al. 2015a; Maćešić et al. 2015b; Maćešić et al. 2016; Čupić et al. 2016b; Čupić et al. 2016c). Here, as in the case of formal kinetics, the main steps in modelling procedure were developed on the BL reaction and then applied to the other systems. Thus, multiple time scale dynamics of the BL reaction model was identified as a source of the mixed-mode oscillations (Fig. 5) and tourbillion mechanism was recognized (Čupić et al. 2013). Complex bifurcations were also found in this model and confirmed by both numerical evidence and theoretical considerations (Stanković et al. 2013; Stanković et al. 2016). Details of the reaction mechanism were studied in the model of the BZ reaction (Fig. 1B) and complex oscillations were also discovered there (Blagojević et al. 2011; Blagojević et al. 2013 and Table 1).

(BG1) $Br^- + HOBr + H^+ \rightarrow Br_2 + H_2O$

(BG-1) $Br_2 + H_2O \rightarrow Br^- + HOBr + H^+$

(BG2) $HBrO_2 + Br^- + H^+ \rightarrow Br_2O + H_2O$

(BG3) $Br_2O + H_2O \rightarrow 2HOBr$

(BG-3) $2HOBr \rightarrow Br_2O + H_2O$

(BG4) $Br^- + BrO_3^- + 2H^+ \rightarrow HOBr + HBrO_2$

(BG5) $2HBrO_2 \rightarrow BrO_3^- + HOBr + H^+$

(BG6) $BrO_3^- + HBrO_2 + H^+ \rightarrow 2BrO_2^\bullet + H_2O$

(BG-6) $2BrO_2^\bullet + H_2O \rightarrow BrO_3^- + HBrO_2 + H^+$

(BG7) $Ce^{3+} + BrO_2^\bullet + H^+ \rightarrow Ce(IV) + HBrO_2$

(BG-7) $Ce^{4+} + HBrO_2 \rightarrow Ce^{3+} + BrO_2^\bullet + H^+$

(BG8) $MK + Br_2 \rightarrow BrMK + Br^- + H^+$

(BG9) $MK + Ce^{4+} \rightarrow Ce^{3+} + P_1 + H^+$

(BG10) $BrMK + Ce^{4+} \rightarrow Ce^{3+} + Br^- + P_2$

(BG11) $MK + Br_2O \rightarrow BrMK + HOBr$

(BG12) $Br_2\,(rast.) \rightarrow Br_2\,(g)$

Table 1. Model BG(1-12) of the BZ reaction consisting of 12 reactions: BG1-BG12. (Blagojević et al. 2008) ***Remark:*** P1 and P2 are the products

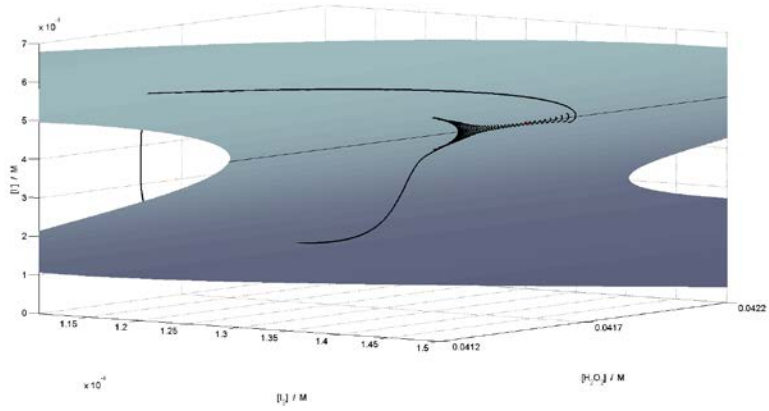


Fig. 5. Critical manifold of the BL reaction model and numerically simulated phase space trajectory over it. Fold line of the critical manifold is also given

Much of our work in the last decade was dedicated to the investigation of the HPA axis, and influences of several perturbing substances on its dynamics (Marković et al. 2011b; Marković et al. 2016; Čupić et al. 2016b; Čupić et al. in press). Due to inherent ultradian oscillations, our model of the HPA axis has a phase sensitive response on perturbations. This property was crucial for successful simulation of numerous effects which were previously unexplained, like different reaction on the same stress during night and day.

2.5 Oscillatory reactions in external fields

The researches of the influence of external fields were commenced in 2014 (Stanisavljev et al. 2004; Stanisavljev et al. 2005). This investigation persists until the present day. So, the influence of MW (2.45 GHz) and RF (30-120 MHz) electromagnetic radiation on the oscillatory reactions was examined at the constant bulk temperature (Stanisavljev et al. 2006; Stanisavljev et al. 2007; Stanisavljev et al. 2011; Stanisavljev et al. 2014). It was found that highly absorbed microwaves decrease the number of oscillations where as reaction dynamics is not affected by RF radiation.

3. Perspectives

Belgrade group is continually spreading its interests in the area of new oscillating reaction systems, new experimental and theoretical techniques, and it keeps producing new insights in the reaction mechanism underlying complex dynamical states. The new contributions to the understanding of the intermittent behaviour in the BL reaction can be expected. Further progress in modelling HPA axis under basal and stress conditions is also in front of us.

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Извод

Доприноси Београдске групе изучавању осцилаторних реакција

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Резиме

Осцилаторна динамичка стања, као облик самоорганизације нелинеарних система, могу се наћи у готово свим наукама, као што су механика, физичка хемија или биомедицина. Иако је порекло ових осцилација различито, тешкоће у моделирању осцилаторних феномена су заједничке на свим пољима. Од 1979. године истраживачи Београдске групе систематски истражују осцилаторне реакције. Како је стабилност устаљених стања кључни проблем у моделирању осцилаторних реакција, у последњих 10 година они су за ту намену усвојили и унапредили моћну технику Анализе стехиометријских мрежа. Затим је идентификовано више типова бифуркација у неколико модела осцилаторних реакција. Чак су и веома сложена хаотична кретања у концентрационом фазном простору окарактерисана и квантификована различитим нумеричким техникама. Установљено је да извор осцилација мешаних модова и других уочених сложених облика динамике представљају процеси који се одигравају на различитим временским скалама. Такође су развијене и аналитичке примене осцилаторних реакција.

Кључне речи: осцилаторне реакције, нелинеарна динамика, Анализа стехиометријских мрежа, нумеричке технике за детекцију периодичних и апериодичних динамичких стања, аналитичка детерминација

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