Hindawi Publishing Corporation
International Journal of Photoenergy
Volume 2015, Article ID 653740, 6 pages
http://dx.doi.org/10.1155/2015/653740

*Research Article*

# The *Cortinarius* Fungi Dyes as Sensitizers in Dye-Sensitized Solar Cells

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Received 11 November 2014; Revised 10 December 2014; Accepted 11 December 2014

Academic Editor: Rajaram S. Mane



The dye-sensitized solar cells have been for the first time prepared using *Cortinarius* fungi extracts as a source of sensitizing dyes. The seven species of *Cortinarius* group, collected in the East Poland, have been used to obtain crude extracts of natural dyes used as sensitizing solutions to prepare DSSCs working electrodes. Extracts and sensitized electrodes have been well characterized by UV-Vis spectroscopy measurements. The device sensitized by *Cortinarius sanguineus* extract has been found the most active in photon-to-current conversion process with efficiency $\eta = 0.64\%$, fill factor FF = 65.9%, photocurrent density $J_{SC} = 1.79$ mA/cm$^2$, and photovoltage $V_{OC} = 541$ mV.

## 1. Introduction

Since the first presentation of efficient device by O'Regan and Graetzel in 1991 [1], the dye sensitized solar cells (DSSC) have become a widely studied inexpensive alternative to silicon solar cells [2]. The main advantage of such devices is relatively uncomplicated construction and ability to obtain high efficiencies with no use of expensive materials and Hi-Purity Chemicals. Typical DSSC is built with two sheets of transparent conductive oxide (TCO) coated glass, one of which, the so-called working electrode, is covered with the mesoporous electrode, mostly made of nanocrystalline $TiO_2$, sensitized with a dye (mostly ruthenium complexes). The second TCO glass sheet, the so-called counter electrode, is covered by a thin layer of platinum. These two electrodes are arranged in a sandwich cell and the space between them is filled with a liquid electrolyte containing $I^-/I_3^-$ redox pair solution in organic solvent(s) [2]. The principle mechanism of the DSSC working cycle is straightforward and may be described with five steps of electron movement. The first step is the excitation of an electron from the ground to the excited state of the dye molecule by a photon of incident light. Next, the excited electron is injected from the dye molecule into the conducting band of the mesoporous semiconducting electrode; afterwards the electron is transported across the mesoporous electrode and external circuit into the counter electrode. Now electron reduces the oxidized form of the redox couple in the electrolyte and, as the last step, the reaction between the reduced form of the redox mediator and the oxidized dye molecule occurs; thereupon the ground state of the dye molecule and oxidized form of the redox couple are restored and the DSSC working cycle is closed [2–4]. In the ideal system the whole process takes place without consumption or permanent transformation of any chemical species and theoretically it can occur as long as illumination is present.

Dyes used in DSSC devices belong to various chemical classes. Most intensively studied are synthetic pigments, especially polypyridine complexes of ruthenium [5, 6], but also metal-free molecules are used for this purpose (e.g., oligothiophenes, coumarins, squaric acid derivatives, triarylamine pigments, etc.) [7]. As the synthesis of these compounds requires multistep reaction, their preparation is expensive and far from the principles of environmental-friendly green chemistry. Interesting alternatives to synthetic dyes used in DSSC are natural pigments [8–13]. Many compounds, isolated from plants and animals, meet the requirements of pigments useful for solar cells: they contain chromophores absorbing the light in a wide wavelength range; the hydroxyl or carboxyl groups of these pigments are able to bind

Table 1: *Cortinarius* fungi used for DSSCs preparation.

| Sample | Scientific name | Locality | Habitat |
|---|---|---|---|
| Css1 | *Cortinarius semisanguineus* | Near Osowiec-Twierdza village (NE Poland) | Mixed forest (*Betula pendula*, *Betula pubescens*, *Pinus sylvestris*) |
| Cs1 | *Cortinarius sanguineus* | Near Białowieża village (NE Poland) | Mixed forest (*Pinus sylvestris*, *Quercus robur*) |
| Csp1 | *Cortinarius* sp. (sect. *Telamonia*) | Near Białowieża village (NE Poland) | Mixed forest (*Pinus sylvestris*, *Quercus robur*) |
| Csp2 | *Cortinarius* sp. (sect. *Dermocybe*) | Near Czerlonka village (NE Poland) | Coniferous forest (*Picea abies*, *Pinus sylvestris*) |
| Cc1 | *Cortinarius croceus* | Near Czerlonka village (NE Poland) | Coniferous forest (*Picea abies*, *Pinus sylvestris*) |
| Css2 | *Cortinarius semisanguineus* | Near Czerlonka village (NE Poland) | Mixed forest (*Pinus sylvestris*, *Quercus robur*) |

to $TiO_2$ surface and they are usually quite stable under sun irradiation. Natural dyes are very attractive for DSSC application as they are low-cost, available in large quantities, and sustainable. There are two approaches to the use of natural colorants in DSSC. According to the first, a single isolated and purified compound (or compounds group) is used as a sensitizer, while the second one involves the use of a crude extract for sensitization of the $TiO_2$ layer. Various natural pigment classes are recognized as active components for photosensitization of DSSCs. The most important are chlorophylls, carotenoids, and anthocyanins. The highest efficiencies ($\eta = 3.7$–$4.2$) were detected for the systems containing a semisynthetic chlorophyll derivative and carotenoid (e.g., neoxanthin or $\beta$-carotene) [14]. In the second approach, the highest efficiencies of photosensitization were observed for the extracts from betalains-containing beet root (*Beta vulgaris*; $\eta = 2.2$–$2.7$) [15], azaphilones-producing angkhak rice mold (*Monascus purpureus*, $\eta = 2.3$) [16], and chalcones-containing flowers of Flame of the Forest tree (*Butea monosperma*, $\eta = 1.8$) [17]. Except for *Monascus purpureus*, no other fungi have been used as a source of DSSCs sensitizers; in particular no species belonging to macromycetes have been studied for this purpose.

The genus *Cortinarius* (Agaricales, Basidiomycota) is a large and very diverse group of fungi. The species belonging to genus *Cortinarius* form agaricoid, medium to large, intensively colored basidiocarps. Peculiar group of the *Cortinarius* species are the taxa belonging to the section *Dermocybe* (commonly known as skin-heads). They produce medium-size fruiting bodies with intense, bright colors of various shades of red, orange, yellow, and ocher. In Nordic countries the extracts from *Dermocybe* representatives are traditionally used for dying textiles, especially wool [18]. The pigments isolated from these fungi belong to anthraquinones (emodin, dermorubin, dermocybin), xanthones (dermoxanthone), pyranonaphthoquinones (cardinalin), and chrysones (dermochrysone) [19–23]. Although quinones and quinone-related compounds contain groups that allow their binding to $TiO_2$ surface, they have been rarely studied as natural sensitizers of DSSCs. The only example is the application of henna extract (*Lawsonia inermis*), containing lawsone, a representative of naphthoquinones [24, 25].

In this work a series of DSSCs sensitized by crude extracts of various fungi from the genus *Cortinarius* was obtained and characterized. The photocurrent characteristics of the cells and spectroscopic data of the extracted dyes were determined.

## 2. Experimental

*2.1. Preparation of Dyes Extracts.* The fungi basidiocarps were collected in autumn 2013. Fruiting bodies were placed in electric fruit dryer within 5 h after picking; the drying temperature was lower than 313 K. Dried material was stored at room temperature in the dark. Then 1 g of finely crushed, dried basidiocarps was mixed with 20 mL of methanol. So obtained mixtures were left at 278 K in the dark. After 24 h the solids were filtered off using 0.45 $\mu$m PTFE syringe filters, and the obtained extracts were used to sensitize titania electrodes for DSSCs. The origin of the fungi used in this study is given in Table 1.

*2.2. DSSCs Preparation and Characterization.* All chemicals used were of analytical grade and were used as received without any additional purification. The procedure used for preparation of the titania electrodes was similar to those described elsewhere [26] and was as follows: 3 mL titanium tetraisopropoxide (Aldrich) was added to 13.5 mL of ethylene glycol (Chempur) magnetically stirred at 333 K. The mixture, after addition 12.6 g of citric acid monohydrate (POCh), was heated under stirring at 363 K, until clear. The transparent sol obtained was mixed with 5.6 g P25 $TiO_2$ (Degussa) by grinding in an agate mortar for 1 hour. The viscous titania paste obtained was spread on fluorine doped tin oxide (FTO) conductive glass substrate (Solaronix) using "doctor blade" technique and sintered in air at 723 K for 2 hours. To prepare the working electrodes for DSSCs, titania electrodes were immersed in *Cortinarius* extracts at 278 K overnight. After dye adsorption, the electrodes were washed with absolute ethanol and dried in hot air stream. Platinum film coated FTO was used as a counter electrode. A typical cell was assembled using a 25 $\mu$m thick, hot-melted, ionomeric foil (Solaronix) as a sealant and a spacer between the electrodes and electrolyte (a mixture of 0.6 M 1-propyl-3-methyl-imidazolium

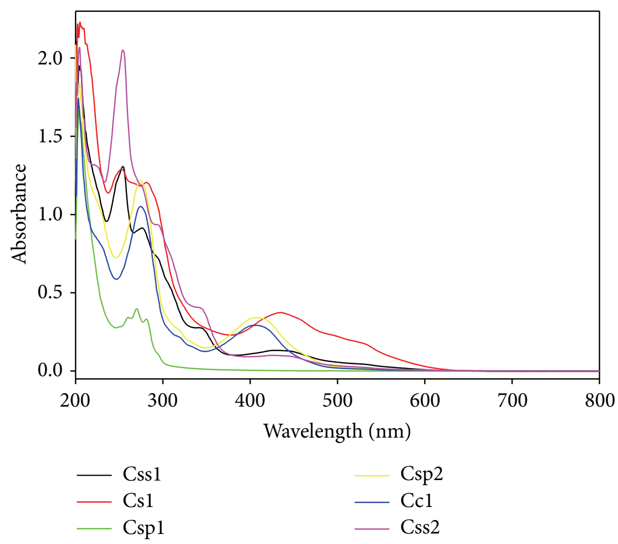


Figure 1: The absorption UV-Vis spectra of *Cortinarius* crude extracts in methanol diluted 30 times.

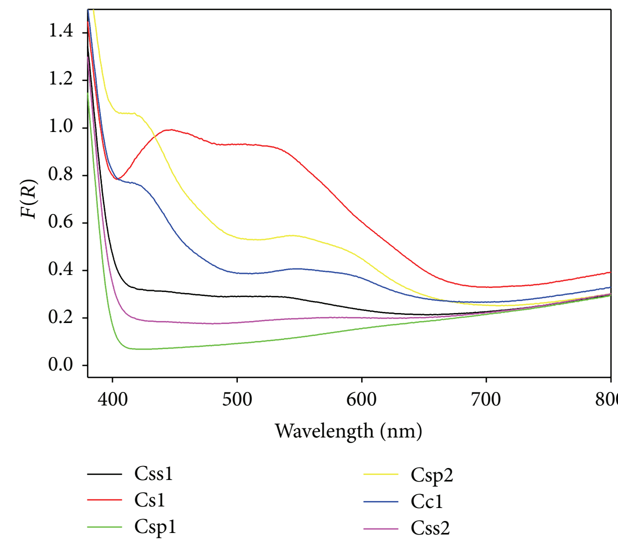


Figure 2: Diffuse-reflectance UV-Vis spectra of sensitized titania electrodes in Vis region.

iodide (Aldrich), 0.03 M iodine (POCh), 0.1 M guanidine thiocyanate (Fluka), and 0.5 M 4-tert-butylpyridine (Aldrich) in acetonitrile (Merck)) have been injected within two holes predrilled in the counter electrode. The final sealing was realized with the use of hot melted sealant and a microscope cover slide. The typical active area of the obtained DSSC was approximately 0.125 $cm^2$. The obtained cells were labeled according to the *Cortinarius* samples names used to obtain sensitizing dye extract.

Photovoltaic characteristics of the cells were measured using a Sun 2000 class A Solar Simulator (Abet Technologies) equipped with an AM 1.5 G filter, with the light intensity adjusted at 1000 $W\cdot m^{-2}$ using a silicon reference cell (ReRa Systems). *J-V* curves were recorded on a Keithley 2400 SourceMeter (Keithley). For each configuration three cells were prepared and photoelectric measurements were repeated five times for every cell prepared, and the shown results are the best ones. The diffuse reflectance UV-Visible spectra (DRUV-Vis) were recorded on a Cary 100 Bio spectrometer (Varian) equipped with 150 mm sphere and absorption UV-Vis experiments were carried out on a Cary 50 Probe spectrometer (Varian).

## 3. Results and Discussion

The most important property of potential sensitizing dye to be applicable in DSSC is its ability to absorb light from the visible region [2]. To examine these properties of the *Cortinarius* fungi crude extracts, the adsorption UV-Vis spectra were measured and the obtained spectra in full UV-Vis range are presented in Figure 1. All presented extracts have strong absorption abilities in UV region and the spectra are very complex, which is most probably caused by the presence of many UV active compounds in all investigated extracts. The visible region of the spectra supports the conclusion about a complex composition of the investigated extracts. The strong visible light absorption abilities are exhibited by 5 of the 6 extracts and only the spectrum of Csp1 extract shows no absorption bands in the visible region, which may have negative influence on the performance of DSSC sensitized by this dye. To get deeper insight in sensitizing abilities of extracted dyes, the diffuse reflectance UV-Vis spectra of titania electrodes sensitized by *Cortinarius* dyes were recorded and are presented in Figure 2. The Kubelka-Munk function curve of sample Csp1 has a shape typical of $TiO_2$ materials (not presented) without any additional absorption bands. The adsorption edge at about 380 nm is characteristic of P25 $TiO_2$ used for DSSC working electrode and is a consequence of a typical, for this material, bandgap value of 3.26 eV [27]. Such a character of Csp1 sensitized electrode may suggest that the cell assembled with its use will not be a very active in photon-to-current conversion process. However, the spectra of the other 5 samples significantly differ in the visible region from those recorded for bare and Csp1 sensitized $TiO_2$. The spectra of samples Css1 and Css2 show a weak broad absorption band between 650 and 415 nm; on the other hand, the spectra of Csp2 and Cc1 samples show two strong adsorption bands with maxima at 555 and 420 nm. The strongest visible light absorption is found for sample Cs1 with adsorption maxima at 525 and 445 nm. The spectra of sensitized electrodes in the visible region also differ from those measured for crude extracts and it is most probably caused by differences in the adsorption abilities of different dyes present in the extracts. It is well known that only the dyes whose molecules have anchoring groups such as –COOH, –$PO_3H_2$, –$SO_3H$, –OH, and so forth may be strongly bonded to the semiconductor surface and give effectively sensitization [2].

The major pigments of *Cortinarius sanguineus* (Cs) are emodin (Figure 3(a)), dermocybin (Figure 3(b)), and dermorubin (Figure 3(c)) [28, 29]. All these components have absorption maxima in the visible region of UV-Vis spectra:

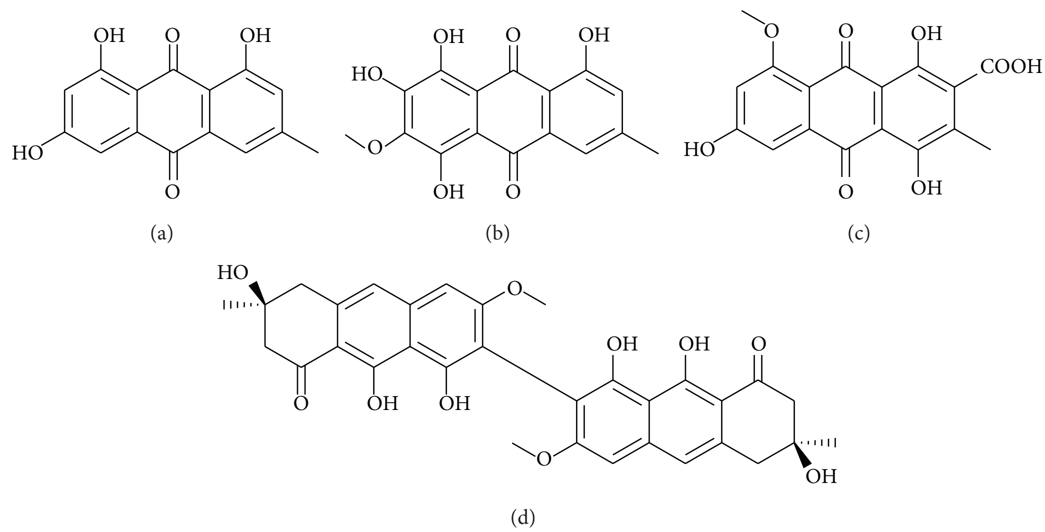

Figure 3: Major pigments of *Cortinarius* sect. *Dermocybe* species: (a) emodin; (b) dermocybin; (c) dermorubin; (d) flavomannin-6,6′-di-O-methyl ether.

the band characteristic of emodin is at 430 nm, while dermocybin and dermorubin give the red-shifted adsorption (*ca.* 490, 520 nm). Comparison of spectrum of Cs extract with the reflectance spectrum of Cs extract adsorbed on $TiO_2$ shows that dermocybin and dermorubin are more effectively sorbed. This is probably a result of the presence of two –OH groups or –OH and –COOH moieties in *ortho* positions in the molecule of these dyes. The same pigments have also been reported to be the major pigments of *C. semisanguineus* (Css), although their concentration in its basidiocarps is lower [28, 29]. This is confirmed by recorded UV-Vis spectra of crude extracts as they show maxima at the same wavelength values for Cs and Css samples. However, for Css, the ratio of the absorbance values for dermocybine (and dermorubin) and emodin is distinctly smaller than for Cs (0.39 versus 0.54), which indicates a smaller relative concentration of dermocybin/dermorubin than emodin. In the UV region of the Css spectra a strong absorption at 252 nm is observed. Comparison of the signal intensities with the values reported for *Cortinarius* pigments indicates that the presence of this band is caused by some component, which is not adsorbing in Vis region. The reflectance spectra of the pigments adsorbed on $TiO_2$ indicate that the sorption of the antraquinone dyes from Css extract is less effective than those from Cs one. This may be a result of competitive sorption of nonsensitizing components, present in Css. In *Cortinarius croceus* (Cc) only minute amounts of antraquinone dyes were detected (no absorption bands >430 nm). The major component ($\lambda_{max}$ = 272 and 401 nm) belongs to chrysone class. The composition of pigment fraction from this species has not been investigated, but one dimeric chrysone dye, flavomannin-6,6′-di-O-methyl ether (Figure 3(d)), has been isolated from their basidiocarps [19, 22]. The reflectance spectra indicate that for Cc, besides the chrysone components, some anthraquinones present in the extract are sorbed on $TiO_2$. The undetermined *Cortinarius* species (Csp2) is very close to *C. croceus*. It is quite plausible that it is this species, although some morphological characters make its identification uncertain.

Table 2: Photoelectrical properties of prepared DSSC devices.

| Sample | $J_{SC}$ (mA/cm$^2$) | $V_{OC}$ (mV) | FF (%) | $\eta$ (%) |
|---|---|---|---|---|
| Css1 | 1.56 | 543 | 63.2 | 0.54 |
| Cs1 | 1.79 | 541 | 65.9 | 0.64 |
| Csp1 | 1.10 | 525 | 64.1 | 0.37 |
| Csp2 | 0.88 | 476 | 62.1 | 0.26 |
| Cc1 | 0.98 | 476 | 64.2 | 0.30 |
| Css2 | 1.52 | 542 | 54.9 | 0.45 |

The photovoltage-photocurrent characteristics curves of *Cortinarius* dyes sensitized devices are presented in Figure 4 and their typical parameters are collected in Table 2. All presented devices show activity in photon-to-current conversion process and the best results (i.e., $J_{SC}$ = 1.79 mA/cm$^2$, $V_{OC}$ = 541 mV, FF = 65.9%, and $\eta$ = 0.64%) were obtained for Cs1 cell. The best performance of Cs1 cell is a natural consequence of the strongest visible light absorption abilities of titania electrode sensitized by Cs1 extract, but it also indicates a relatively good yield of electron injection process from excited dye molecules to the $TiO_2$ conduction band. On the other hand, the poor efficiency of electron injection process may result in surprisingly poor performance of Csp2 and Cc1 devices, whose electrodes show strong light absorption abilities in visible region. It is very difficult to establish the reasons for such behavior of the *Cortinarius* dyes sensitized cells, when crude extract are used. The typical causes of low efficiency of natural dyes in DSSC may be, for example, inadequate energy of the dye excited state, the fast recombination process occurring in the dye molecule, and/or the presence of compounds supporting the recombination processes in the cells, in the extracts [2]. The relatively high efficiency of Csp1 sensitized device is also unexpected,

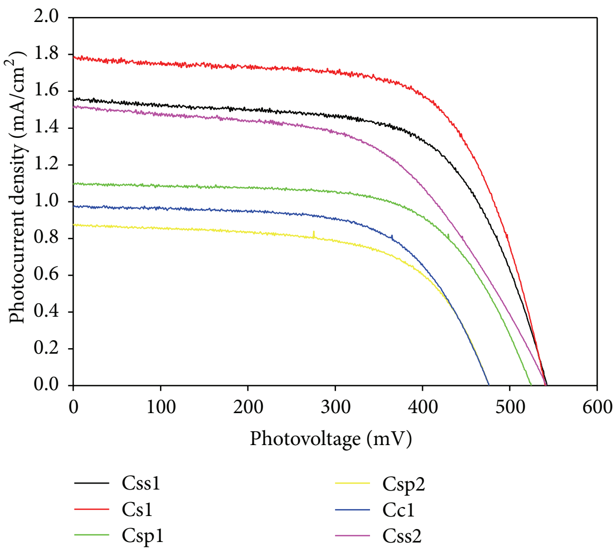


Figure 4: The *J-V* curves of the DSSC devices sensitized by *Cortinarius* dyes.

because of very poor visible light absorption abilities of both crude extract and the sensitized electrode. We speculate that the substances adsorbed on the Csp1 electrode surface may react with other cell components (most probably iodine present in electrolyte) to form new species with good abilities of visible light absorption, but the research for the explanation of this phenomena is continued.

## 4. Conclusions

The *Cortinarius* fungi crude extracts used as sensitizing solutions for preparation of DSSC's working electrodes gave relatively efficient devices in photon-to-current conversion process. The photoconversion efficiency of particular cell types strongly depends on both dye adsorption efficiency and the abilities of visible light absorption of working electrodes. The dye adsorption efficiency is affected not only by the chemical structure of the dye molecules present in the extracts, but also by some probable competitive adsorption of some visible-light-inactive species present in extracts. These preliminary studies show that *Cortinarius* fungi may be good source of sensitizers for DSSCs so further investigation is needed to better understand the dye adsorption and sensitization mechanism.

## Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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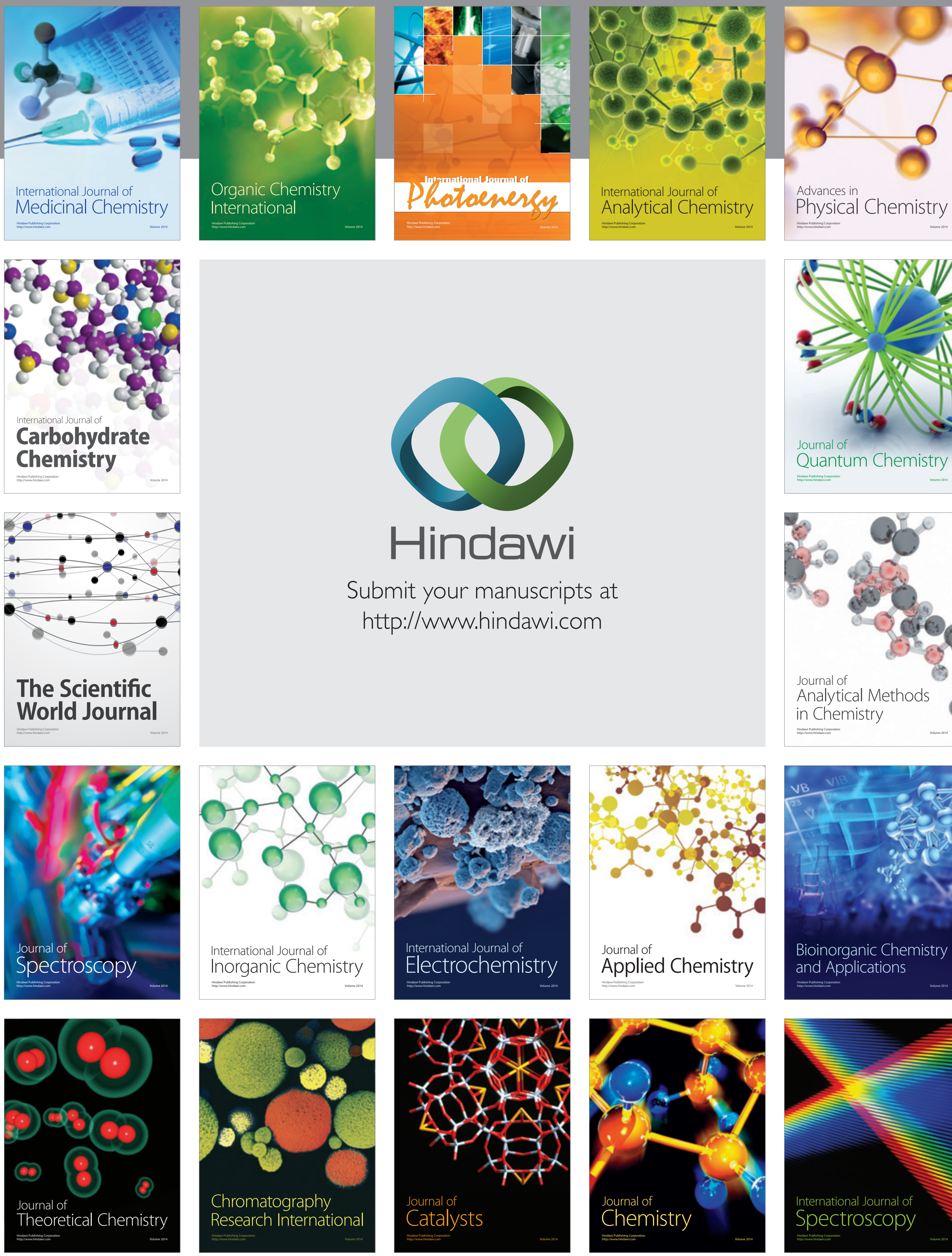
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