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Review

Analytical techniques for steroid estrogens in water samples - A review

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H I G H L I G H T S

- Determination of steroid estrogens has become an important issue in water samples.
- Analyses of steroid estrogens in water require highly sensitive analytical technique.
- Instrumental and non-instrumental analytical technique are use to quantify estrogens.
- ELISA appears to be the most reliable technique to detect estrogens in water samples.

A R T I C L E I N F O

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In recent years, environmental concerns over ultra-trace levels of steroid estrogens concentrations in water samples have increased because of their adverse effects on human and animal life. Special attention to the analytical techniques used to quantify steroid estrogens in water samples is therefore increasingly important. The objective of this review was to present an overview of both instrumental and non-instrumental analytical techniques available for the determination of steroid estrogens in water samples, evidencing their respective potential advantages and limitations using the Need, Approach, Benefit, and Competition (NABC) approach. The analytical techniques highlighted in this review were instrumental and non-instrumental analytical techniques namely gas chromatography mass spectrometry (GC-MS), liquid chromatography mass spectrometry (LC-MS), enzyme-linked immuno sorbent assay (ELISA), radio immuno assay (RIA), yeast estrogen screen (YES) assay, and human breast cancer cell line proliferation (E-screen) assay. The complexity of water samples and their low estrogenic concentrations necessitates the use of highly sensitive instrumental analytical techniques (GC-MS and LC-MS) and non-instrumental analytical techniques (ELISA, RIA, YES assay and E-screen assay) to quantify steroid estrogens. Both instrumental and non-instrumental analytical techniques have their own advantages and limitations. However, the non-instrumental ELISA analytical techniques, thanks to its lower detection limit and simplicity, its rapidity and cost-effectiveness, currently appears to be the most reliable for determining steroid estrogens in water samples.

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Contents

1. Introduction	359
2. Discussion	360
2.1. Analytical procedures	360
2.2. Sample preparation	360
2.3. Analytical techniques	361
2.4. Need, approach, benefit, and competition (NABC)	361
3. Future trends	366
4. Conclusions	366
References	367

1. Introduction

Endocrine disrupting compounds (EDCs) have received considerable attention worldwide due to their potential as environmental contaminants capable of interfering with the normal functions of the endocrine system (Yang et al., 2011). A wide variety of contaminants have been recognized as EDCs, including plasticizers, surfactants, pesticides, as well as natural and synthetic estrogens (Esperanza et al., 2007). Among the large group of EDCs, steroid estrogens are the major contributors to endocrine disrupting activity in environmental samples (Vulliet et al., 2007), and are of particular interest due to their high estrogenic potency with physiological active concentrations ranging from picograms per liter (pg/L) to nanograms per liter (ng/L) (Farre et al., 2007).

Steroid estrogens are a group of biologically active compounds, which derive from cholesterol and have a cyclopentan-*o*-perhydrophenanthrene structure in common (Cui et al., 2006). Estrogens are predominantly female hormones that maintain the health of reproductive tissues, skin, breast, and brain (Manickum and John, 2014). Natural estrogens such as estrone (E₁), estradiol (E₂) and estriol (E₃) are secreted by the adrenal cortex, ovary, testis, and placenta in humans and animals, while synthetic estrogens such as ethinylestradiol (EE₂) are the main components in oral contraceptive pills and hormone therapy (Cui et al., 2006; Zhang et al., 2011; Ye et al., 2012). All humans and animals urinary and fecal excretions contain both natural and administered synthetic estrogens which therefore potentially end up in the environment through sewage discharge or animal waste disposal (Lintelmann et al., 2003; Atkinson et al., 2012; Ye et al., 2012). Wastewater with incomplete removal of estrogens from treatment systems and direct discharge into receiving water will lead to major sources of estrogenic pollution in water samples (Yang et al., 2011). The reason for the occurrence of steroid estrogens in water samples is because water is a good carrying medium for polar and semi-polar compounds, and they are closely related to each other (Bialk-Bienlinska et al., 2016). The physicochemical properties showed steroid estrogens dissolve poorly in water, as natural estrogens have a solubility of about 13 mg/L and synthetic estrogens have a solubility of around 4.8 mg/L (Lai et al., 2000). Furthermore, steroid estrogens have a low volatility, as shown by their very low vapor pressure ranging from 2.3×10^{-10} to 6.7×10^{-15} mmHg (Lai et al., 2000). These characteristics made steroid estrogens less frequently found in water samples and have greater tendency to partition to sediments (Combalbert and Hernandez-Raquet, 2010). Moreover, the octanol-water partition value (K_{ow}), which varies between 2.8 and 4.2, indicates that steroid estrogens are hydrophobic organic compounds (Lai et al., 2000). These values indicated that steroid estrogens have low dissolution in water and prefer sorption on soil and sediment (Ying et al., 2002). Ultra-trace levels of steroid

estrogens in water samples have become a growing concern over the recent years, because of their potential risk of interference with reproduction and development in both humans and animals (Briciu et al., 2009). Analytical methods, namely instrumental and non-instrumental analytical techniques have therefore been developed for the determination of steroid estrogens in water samples. (Swart and Pool, 2007; Mispagel et al., 2009; Yang et al., 2011; Atkinson et al., 2012; Ye et al., 2012; Muz et al., 2012; Zhou et al., 2012; Huang et al., 2014; Manickum and John, 2014; Pessoa et al., 2014).

There are already several existing reviews related to the analysis of steroid estrogens in water samples (Alda and Barcelo, 2001; Ying et al., 2002; Pacakova et al., 2009; Briciu et al., 2009; Cajthaml et al., 2009; Combalbert and Hernandez-Raquet, 2010; Pereira et al., 2011; Silva et al., 2012; Manickum and John, 2015). Ying et al. (2002) and Combalbert and Hernandez-Raquet (2010) reviewed the occurrence, fate and biodegradation of steroid estrogens in wastewater and manure. Silva et al. (2012) reviewed the elimination processes of E₁, E₂, E₃, and EE₂ from wastewater whereas Cajthaml et al. (2009) only focused on the elimination of synthetic estrogen EE₂. Pereira et al. (2011) studied the occurrence and removal of steroid estrogens from drinking water by analyzing disinfection by-products. The analytical procedures for the analysis of steroid estrogens in water samples were reviewed by Alda and Barcelo (2001), Pacakova et al. (2009), and Briciu et al. (2009), who specifically focused on analytical techniques such as gas chromatography, liquid chromatography, and enzyme-linked immunosorbent assay. In addition, Manickum and John (2015) reviewed enzyme-linked immunosorbent assay as the preference technique to quantify steroid estrogens in wastewater, which focused in South Africa. Moreover, Rayco et al. (2014) reviewed various liquid chromatography methods including tandem mass spectrometry, for steroid estrogen analysis in water samples. All previous reviews have focused on analytical procedures and more specifically on occurrence, fate, biodegradation, and elimination of steroid estrogens in water samples. However, there is still a lack of reviews devoted to the advantages and limitations of both instrumental and non-instrumental analytical techniques available for the determination of steroid estrogens in water samples such as wastewater, river water, drinking water, tap water, underground water, and well water. Selection of the most reliable analytical techniques for determining steroid estrogens in water samples remains therefore a challenge. A critical review concerning the most reliable analytical techniques would lead to further optimization of, the methodologies involved, which remain costly and time consuming, ensuring both time-saving and cost efficiency.

The objective of this paper was to present an overview of both instrumental and non-instrumental analytical techniques available for the determination of steroid estrogens in water samples.

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