SYNTHESIS AND CHARACTERIZATION OF SELENIUM NANO PARTICLES BY HIGH ENERGY BALL MILLING (HEBM) TECHNIQUE

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ABSTRACT

In present day, supplementation of extra minerals and vitamins is highly essential in commercial diets due to high productivity and to withstand the detrimental effects of different stresses. Selenium is one of essential trace minerals for better growth and productivity as well as anti-stressor in commercial broilers. Nano-selenium can effectively be synthesized through High Energy Ball Milling (HEBM) technique from its precursor, for use in commercial broilers as anti-stressor and to support multiple bodily functions. The prepared nano particle had 44.5 % of selenium as measured by Energy Dispersive X-ray (EDAX) analysis with the product yield of 50 g/hr. The chemical composition of sodium selenite powder was same as that of the original mega particle. The size of Se nano particle ranged from 37-85 nm as analyzed through Field Emission Scanning Electron Microscope (FESEM). X-Ray diffraction pattern confirmed that the synthesized Se nano particle was free of impurities and provided accurate information on the atomic arrangements. The Fourier Transform Infra-Red (FTIR) spectrum of synthesized nano particle source of selenium peaks was located at 3023.26, 2800.12, 2502.23, 2314.17, 1610.40 and 1413.30 cm-1 which showed chemical bonding in a target material. The zeta potential of nano selenium was -23.30 mV when analyzed through particle size analyzer. Se nano-particles could be successfully synthesized through High Energy Ball Milling method from its precursor and could be characterized for its quantity, size, shape, stability and purity. The synthesized Se nano-particles could be utilized for the conduct of biological trial in commercial broilers.

Key Words: Concentration, Nano Se, Particle Size Purity, and Zeta potential

INTRODUCTION

The faster industrialization and improved productivity of recent day commercial poultry, obviously face several constraints in production and productivity in the arena of global warming. Hot environment will substantially reduce productivity of poultry especially broilers. Supplementation of minerals and vitamins are highly essential to overcome the deleterious effects of hot environment and to maintain better production potential with proper immunity.

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Recently trace minerals in the form of nano-particles are effectively used to fulfill the requirement of minerals in the poultry diets. The extreme smaller sized nano particles has unique physical properties and different when compared to their conventional forms. Nano form of supplementation increases the surface area and leads to increased rate of absorption and utilization of minerals resulting in reduction of required quantity and feed cost. Selenium is one of the essential minerals required for optimal growth and productivity by supporting multiple functions related to poultry production, fertility and disease prevention.

With the recent development of nanotechnology, nano-selenium (nano-Se) has attracted widespread attention because nanometer particulates exhibit novel characteristics such as large surface area, high surface activity, high catalytic efficiency, strong adsorbing ability and low toxicity (Wang *et al*., 2007; Zhang *et al*., 2008). This nano-selenium has to be synthesized by simple and efficient technology for use in commercial poultry as anti-stressor or productivity enhancer. Hence, the present study was undertaken to synthesize and characterize selenium nano particles through High Energy Ball Milling (HEBM) technique compared to other conventional methods for utilization in biological trial with commercial broilers.

MATERIALS AND METHODS

Synthesis of nano-selenium

High Energy Ball Milling (HEBM) technique was adopted to prepare nano particle source of selenium. Nano particle source of selenium was synthesized by grinding feed grade sodium selenite in a ball mill (8000D Mixer/Mill – Dual High Energy Ball Mill) @ 1060 cycles/min using two 12.7 mm stainless steel ball in each jar of 75 ml capacity for 60 mins (Munoz *et al*., 2007). The percentage of selenium present in prepared nano particle was measured by Energy Dispersive x-ray analysis (EDAX) following the protocol outlined by Russ (1970).

Characterization of nano-selenium

The size of the synthesized selenium nano particle was determined by adopting the procedure demonstrated by Yao and Kimura, (2007) using Field Emission Scanning Electron Microscope (FESEM). The prepared nano particle cold source was employed and was placed on a carbon coated Cu grid and examined using an FE-SEM (Carl Zeiss SUPRA-55).

Structural aspects of prepared selenium nano particle were determined by X-Ray Diffraction technique using Rigaku Mini Flex-II Desktop X-Ray Diffractometer as per the protocol explained by Theivasanthi and Alagar (2010).

The surface chemistry and functional group of the synthesized selenium nano particle was investigated by Fourier Transform Infra-Red (FTIR) spectroscopy (Agilent Model Cary 630) according to the method of Chattopadhyay *et al*. (2014).

Zeta potential (mV) of the prepared nano particle was determined based on the principle of photon correlation spectroscopy using Particle Size Analyzer (HORIBA Scientific nano partica SZ-100).

RESULTS AND DISCUSSION

The product yield, particle size, zeta potential, mineral content of nano particle of selenium produced by high energy ball milling are presented in Table 1. The percentage of selenium was 44.5 per cent in prepared nano particle as measured by Energy Dispersive X-ray (EDAX) analysis. The product yield of nano particle source of selenium produced by physical method using ball mill was 50 g/hr. Nano Se particles were successfully produced by using High Energy Ball Milling technique through physical method of synthesis. Similarly, McCormick and Froes, (1998) produced large quantity of nano-particles with low production cost by using mechanical milling technique. High Energy Ball Milling technique is the simple and inexpensive method of production of nano-particles of different materials as stated by El-Eskandarany, (2001). The same technique was also adopted to produce iron and zinc nano-particles by Munoz, (2007) and Salah *et al*. (2011) respectively with sufficient quantity to obtain ideal size of nano-particles. Hence, High energy ball milling is one of the physical methods in synthesis of nano-particles with various shapes and dimensionalities with low production cost. Based on the milling time, the structural and chemical reactions occurred in the minerals and the force applied during milling leads to fracture of the particles which resulted in required quantity and quality of nano-particles.

The size of Se nano particle analyzed through Field Emission Scanning Electron Microscope (FESEM) was less than 100 nm. The FESEM image of produced nano particle source of selenium was determined between the sizes ranged from 37-85 nm (Fig. 1). Similarly, Yao and Kimura, (2007) mentioned FESEM is a useful tool for high resolution surface imaging in the field of nanomaterial sciences for magnification of gold nano-particles. Silver nano-particles with the size of 10 and 25 nm had been measured successfully by Kaviya *et al*. (2011). FESEM might be the well-defined technique to study the size and shape in 3D nano-particles superlattices due to its higher resolution when Field Emission source is combined with Scanning Electron Microscope.

Table 1 Characterization of nano particle source of selenium

S. No.	Characterization parameters	
1	Chemical name of source	Sodium selenite
2	Mean product yield (g/hr)	50
3	Size (assessed through FESEM) nm*	37-85
4	Zeta potential (mV) *	-23.30
5	Selenium content assessed through EDAX (%)*	44.5

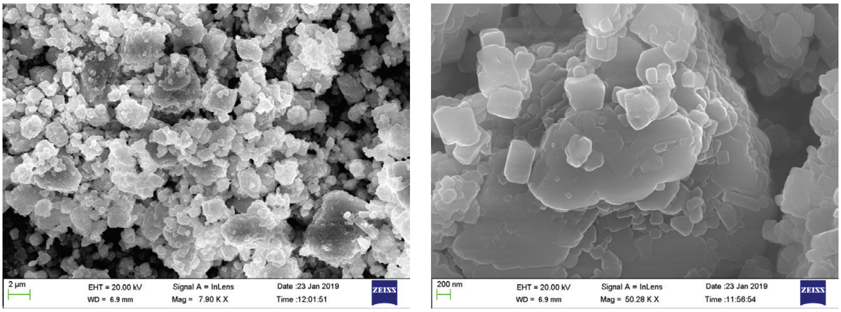

Fig. 1 Field Emission Scanning Electron Microscope (FESEM) image of synthesized nano particle source of selenium

The X-Ray diffraction (XRD) pattern of synthesized nano particle source of selenium is shown in Fig. 2. The data recorded in the 2θ was analyzed using Jade 6.0 software. X-Ray diffraction pattern confirmed that the synthesized Se nano particle was free of impurities as it did not contain any characteristic XRD peaks other than selenium peak and the samples were nano in nature. The same analysis was conducted by Sharma *et al*. (2012) for crystallographic characteristics of bulk nano and thin film material to study the structural dimension of nano-particles. Similarly, Yadav and Bajpai, (2017) obtained the XRD pattern of nano powder synthesized at pH 9.5 with pure CuS phase without any impurity. The intensities based on XRD pattern confirmed that the synthesized Se nano-particles were free from impurities and provided accurate information on the atomic arrangements.

The typical FTIR spectrum of synthesized nano particle source of selenium peaks was mainly located at 3023.26, 2800.12, 2502.23, 2314.17, 1610.40 and 1413.30 cm^{-1}. The shift in peak from 3023.26 to 2800.12 cm^{-1}corresponded to OH/C-C bending which might be responsible for reduction. The functional groups of synthesized Se nano-particles were analyzed by Fourier Transform Infra-Red (FTIR) spectroscopy which showed

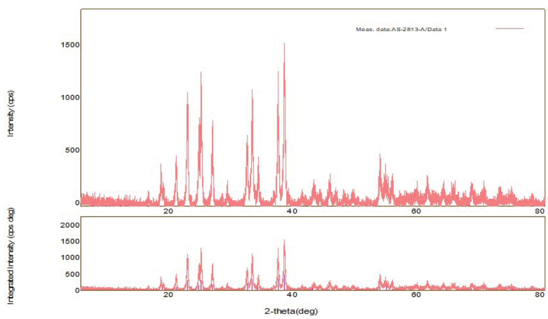

Fig. 2. X-Ray diffraction (XRD) pattern of synthesized nano particle source of selenium

chemical bonding in a target material. In accordance with this Yao and Kimura (2007) stated that FTIR was effectively utilized to study the functional group of gold nano-particles. Similarly Baudot *et al*. (2010) unveiled the identity of functional group of Carbon nano-particles through FTIR and suggested that FTIR is the best tool to study the chemical bonding of nano-particles. Kaviya *et al.* (2011) observed the shift in the absorption band after bio-reduction at $1601 cm^{-1}$ to $1584\ cm^{-1}$ and indicated the formation of nano-particles. The appearance of this peak was due to the presence of hydroxyl group stretching vibration of phenolic compounds which was responsible for the formation and stabilization of synthesized nano-particles.

The chemical composition of sodium selenite powder was analyzed by Energy Dispersive X-Ray (EDAX) analysis and shown in Fig. 3. This confirmed the presence of 44.5 per cent selenium content which was same as that of the original mega particle. Aparna and Karunakaran (2014) produced nano selenium powder with 94.69 per cent selenium content from 99 per cent pure selenium powder. Similarly Arulnathan *et al*. (2016) produced nano selenium with 98.57 per cent selenium from 99 per cent pure selenium powder. However, Jimeno-Romero *et al*. (2016) stated that the chemical composition of nano-particles depended on size and biological effects. They obtained accurate Se content of nano-particles through HEBM method and resulted in no loss of minerals. The accuracy of selenium content was mainly due to nano-particles size and biological effects that gave a clear picture about a cellular structures present in the nano-particles.

The zeta potential of nano selenium synthesized in this study was -23.30 mV when analyzed through particle size analyzer. Zeta potential is an important parameter to study the stability of nano-particles. The zeta potential of +30 to -30 mV considered to have high degree of stability was suggested by Xu (2008) and Clogston and Patri (2011) in anionic and cationic nano-particles. Similar to the result of this study, Nanocomposix, (2012) also suggested zeta potential of -25 to +25 mV had high degree of stability. The Se nano-particles produced in this study had high degree of stability due to Van Der Waal Inter-particle attractions as suggested by Nanocomposix, (2012).

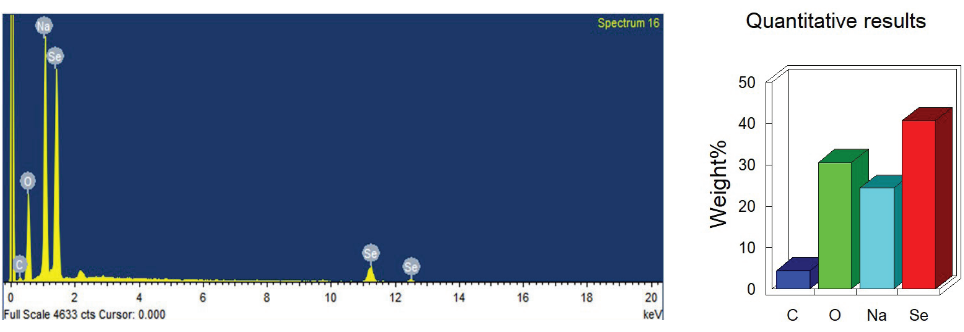

Fig. 3. EDAX spectrum of Se nanoparticles

Se nano-particles could be successfully and effectively synthesized through High Energy Ball Milling method from its precursor and the same could be characterized for its quantity, size, shape, stability and purity. The synthesized Se nano-particles could be utilized for the conduct of biological trail in commercial broilers.

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