which allows the regulation of the size of nanoparticles by manipulating irradiation parameters [22], methods of hydrothermal synthesis [19] and sonochemical reduction [14,20], as well as by using wet reduction methods [23–32]. Wet reduction methods characterized by the formation of nanoparticles of silver on surfaces of mineral matrices involve mineral matrices being impregnated with an aqueous solution of AgNO₃ and the subsequent reduction of silver ions to metallic silver. As a reducing agent, various compounds such as D-glucose [23], ascorbic acid [24], sodium acrylate [25], sodium borohydride [12–15,26–28], sodium citrate [20,28], aspartic acid [29], tanning acid [30], poly(vinylpyrrolidone), carboxymethyl cellulose and gelatin [31] are used. It has also been reported that the formation of silver nanoparticles could be carried out by a reduction in ethanol solution in the presence of certain surfactants [32], as well as with hydrazine dihydrochloride in a slightly alkaline solution [33] or in an acidic medium with sodium formaldehyde sulfoxylate [34]. Sodium borohydride remains the most commonly used reducing agent. Layered clay minerals were used to host silver nanoparticles in the interlayer space and to enhance their bond to the carrier surface. Pre-intercalation of the interlayer space by high molecular weight compounds, such as polyacrylonitrile [15] and dimethylsulfoxide [12], was carried out. Graphene oxide coating was used to improve AgNP stability in the diatomite composite [10].

The use of mineral matrices as carriers of silver nanoparticles can be promising from the environmental and economical points of view. The practical applications of such composites may be various. Silica-based composites are used as an adsorbent for purifying water from mercury ions [6], in catalytic systems [7] and as antibacterial biofilms [8]. Montmorillonite-based nanocomposites (Ag/MMT) were applied in biopolymer matrices for packaging materials [16]. Diatomite coated with silver nanoparticles is used for antimicrobial water treatment [11,35]. Silver-containing composites based on zeolites [17,18] and diatomite [10,11] showed active antibacterial properties.

Therefore, the main purpose of this study was, firstly, to obtain a hybrid composite (AgCl, Ag)NPs/diatomite) based on natural diatomite and silver nanoparticles in the form of metallic silver (AgNPs) and silver chlorides (AgCl-NPs) simultaneously and, secondly, to investigate the antibacterial potential of obtained composites against Gram-positive (*Staphylococcus aureus*) and Gram-negative (*Klebsiella pneumoniae*) bacteria. In the present experiment, the chloride ion source was the mineral impurity halite, naturally present in the diatomite support. From an application perspective, the obtained formulations can be found as bioactive and biocompatible materials in the medicine, cosmetic or food industry (packaging materials).

2. Materials and Methods

2.1. The Diatomite Sample

The source of natural diatomite was the Mugodzhar deposit (Kazakhstan, Aktobe region). The diatomite samples were prepared by hand-crushing in a porcelain mortar with subsequent sieving to obtain a granulometric fraction with particle size < 0.30 mm.

2.2. Characterization Methods and Instrumentation

The elemental composition of natural diatomite and the sizes and distribution of nanoparticles in synthesized (AgCl, Ag)NPs/diatomite composite structures were determined by using scanning electron microscopy (SEM, LEO 1430 VP, Leo Electron Microscopy Ltd, Cambridge, United Kingdom) coupled with an energy dispersive X-ray (EDX) detector (XFlash 4010, Bruker AXS, Bremen, Germany) and transmission electron microscopy (TEM, FEI Tecnai F20 X-Twintool, FEI Europe, Frankfurt/Main, Germany). The X-ray powder diffraction (XRD) method was used to determine the mineral composition of natural diatomite, and the formation of Ag nanoparticles was examined using a Philips X 'Pert Pro diffractometer (XRD, Malvern Pananaliytical Ltd, Malvern, United Kingdom) with Cu-Kα-radiation (γ = 0.1541 nm, 40 kV, 30 mA). The XRD pattern data were collected over an angular range of 5–100°2θ with step sizes of 0.01. Functional groups of diatomite samples were determined on an FTIR spectrometer (FTIR ATR, Vertex 70, Bruker Optics, Bremen, Germany) equipped with a DLatTGS detector. The FTIR spectra were recorded by averaging 64 scans