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Review

An Overview of Micro- and Nano-Dispersion Additives for Asphalt and Bitumen for Road Construction

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Abstract: The main motivations for the development of research in the area of appropriate additives for asphalt and bitumen are the enhancement of their properties and improvement of their production process, including the reduction in environmental burden. Many additives improve the properties of mineral–asphalt mixtures. Traditionally, additives such as the following are applied: elastomers, plastomers, latexes, rubber powder, resins, and others. Currently, the modification of asphalt and bitumen materials by traditional additives can be replaced by nanomaterials that better fit the requirements of modern industry. New solutions are required, which has led to years of studies researching micro- and nano-additives. The main aim of the article is to analyze contemporary research where micro- and nano-additives were applied to asphalt and bitumen and summarize the advantages and disadvantages of the implementation of these additives for road construction. The article studied the state of the art in this area based on the literature research. It presents the possible materials' solutions, including their properties, used technology, and featured trends for road construction. The challenges for further projects are discussed, especially environmental issues.

Keywords: micro-additive; nano-additive; asphalt; bitumen; nanocomposite; road construction

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1. Introduction

Nanoparticles have a significant impact on different material properties if they are selected properly and implemented correctly in the matrix. Even a small amount of nanoparticles could significantly change the material's properties, including increasing mechanical properties, improving processing properties, or even giving new properties to the materials [1,2]. Nano-additives have found application in many areas of human activities, including civil engineering, machine design, medicine and others. Their application can be also an answer to the trends connected with the reduction in environmental burden [1,3]. Despite their applications in many areas, new research on nanocomposites is required, which should include not only further improvements in technology and the design of the new materials but also be focused on the safety applications of nanoparticles and long-term properties of obtained materials [2,4]. Another important element is the investigation of the nature of the distribution of nanoparticles in the volume of the material [5,6]. The dispersion and distribution of these is a valuable observation from a technological point of view [7,8].

The main motivation for the development of the research in the area of appropriate additives and technological processes that will improve the properties of asphalt and bitumen mixtures and reduce their production temperature is the increasing demand for higher-quality roads and also trends connected with reducing their environmental burden [9,10]. In this article, the terms of "asphalt" and "bitumen" do not have an equal meaning. The bitumen, in this case, is understood as one of the constituents (binder) in an asphalt mixture [11–13].

In the literature studies, many additives improve the properties of mineral–asphalt mixtures. Traditionally, additives such as the following were applied [14,15]:

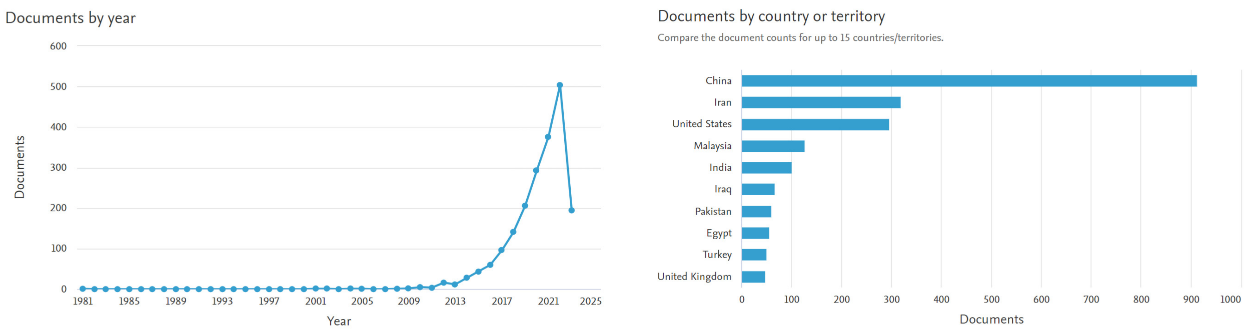
- Elastomers (including styrene–butadiene–styrene (SBS) copolymer, styrene–isoprene–styrene SIS copolymer, styrene–butadiene (SB), and others);
- Plastomers (i.e., ethylene–vinyl acetate EVA copolymer, polyisobutylene PIB and others);
- Latexes (such as chloroprene CR, butadiene–styrene SBR and others);
- Rubber powder;
- Resins (including: epoxy resins and polyester resins);
- Thermoplastics and polyolefins (such as: polypropylene PP, high and low-density polyethylene HDPE/LDPE).

Currently, the modification of asphalt and bitumen materials by traditional additives can be replaced by nanomaterials that better fit the requirements of modern industry. New solutions are required. Because of that, research using micro- and nano-additives has been conducted for several years [16–18].

The main aim of the article is to analyze contemporary research where micro- and nano-additives were applied to asphalt and bitumen and summarize the advantages and disadvantages of the implementation of these additives for road construction. The article briefly presents the overall knowledge in the area of nano-additives and next studies the state of the art in the area of using these materials in road construction branches based mainly on the literature research. It presents the possible materials solutions, including their properties, used technology, and featured trends for road construction. Finally, the challenges for further projects are discussed, especially environmental issues.

2. Methods

The systematic review was made using Scopus (ScienceDirect) as a main search tool and as supporting tools for the following databases: ACS Publications, Wiley Online Library, IEEE Xplore Digital Library and Google Scholar. Additionally, research has taken into consideration patents (Google Patents and EUIPO) and the databases of the standards, especially series EN (ITEH STANDARDS). The used keywords were the combination: "asphalt" (or "bitumen") and "additive" and "nano". The results show 1987 records in the database (Figure 1). They were checked, and the most relevant documents were selected for this report.



(**a**) (**b**)

Figure 1. Results of the search in the Scopus database: (**a**) published documents by year; (**b**) published documents by country [19].

The analysis of the results shows that the topic is very new. The first publication was in 2008, but in fact, rapid growth started after 2013. Today, the interest in this topic is developing very fast. The analysis of countries shows that this topic is crucial for developing countries, especially China, as well as developed ones—such as the United States.

The knowledge from the literature has been supplemented by the authors' microstructure investigation of nanoparticles. They are presented for better visualization of the problem for the reader. The microstructure images have been taken by using scanning electron microscopy (SEM), type JEOL JSM-IT200 (JEOL, Tokyo, Japan). Before the observation, samples were placed on a stand carbon pot and covered with a layer of gold (DII-29030SCTR Smart Coater, JEOL, Tokyo, Japan) to ensure proper conductivity.

3. Nanocomposite Materials

3.1. Classification

Nanocomponents can be classified according to different criteria, such as dimensions, form, origin, morphology, applications, materials properties, manufacturing process, and others [3,4]. Nanocomponents can have different forms, depending on the application, such as [20]:

- Circles, including nanoparticles, fullerenes, and quantum dots;
- Fibers, nanotubes, wires, rods, and other linear structures;
- Thin films, layers, sheets, plates, and similar;
- Bulk structured nanomaterials, for example polycrystals.

The presented classification according to the forms can be treated as basic. It is worth noting, however, that some authors distinguish additional forms of nanoparticles [21–24]. One of these possible forms is fused fractal aggregates when elementary nanoparticles form branched chain-like structures. An example of the nanomaterial that appeared in this form is fumed silica—nano-silica [21,22]. The fused fractal aggregates are treated as an intermediate form between circular nanoparticles and bulk-structured nanomaterials [23,24].

Today, among these forms, the nanoparticles seem to be the most widely and most universal. They find applications in various matrices and are used for different purposes. The most popular functions for nanoparticles are connected with influencing the antibacterial properties, mechanical properties, conductivity and thermal properties, magnetic properties, and processing properties, among others [2,25]. Also, they may be manufactured from many materials, which significantly influences the composite's properties and area of application [26,27]. In the literature, there are a lot of classifications for nano-materials: one of the most frequently applied is presented in Figure 2.

Organic nanoparticles are a wide group that includes very different materials, including nanoparticles obtained with many synthetic polymer materials as well as others, such as nano-cellulose [28]. Today, polymer nanoparticles are based on synthetic polymers, including PCL—poly(ε-caprolactone), PEG—poly(ethylene glycol), PLA—poly(lactic acid), and PLGA—poly(lactide-co-glycolide) [29]. They are used not only in the traditional form of spheres but also as a nanocapsule: for example in drug delivery systems. In this group, there are also other materials, including bio-based, such as nano-cellulose. It has a lot of applications, including biological sensors for advanced applications in wastewater and desalination technologies [3,28].

The second group is inorganic nanoparticles, which can be divided into some types. Among others, these include nanoparticles of pure metals, for example, silver and copper [3,30,31], and metal oxides, such as titanium dioxide (TiO_2), alumina oxide (Al_2O_3), and zinc oxide (ZnO). These two groups are widely investigated as additives for different materials. In this group, from the point of view of the application in asphalt and bitumen materials, there are very important ceramic and mineral nanoparticles, especially nano-silica, nano-clay, nano-hydrotalcite, and montmorillonite [32,33]. These nanoparticles also find application in building materials, including road construction [33,34], where they usually enhance physical and rheological properties as well as the durability of the matrix [31]. These kinds of nano-additives also can improve wear resistance, fatigue properties, and thermal properties, including improving the high-temperature performance of materials.

The most widely investigated are in this case nano-silica and nano-clays [2,31]. Additionally, the particles in the group 'inorganic' have an antibacterial effect [3], especially various metal and metal oxides [35]. They are also employed for catalytic applications, very often as polymer/metal oxide hybrid nanoparticles for water and air purification [3,36]. The important challenge in the case of inorganic nanoparticles is their poor affinity to organic materials such as bituminous and polymers, which leads to particle aggregation [37,38]. Because of that, inorganic particles are often modified by surface functionalization or the addition of surfactants [13,39].

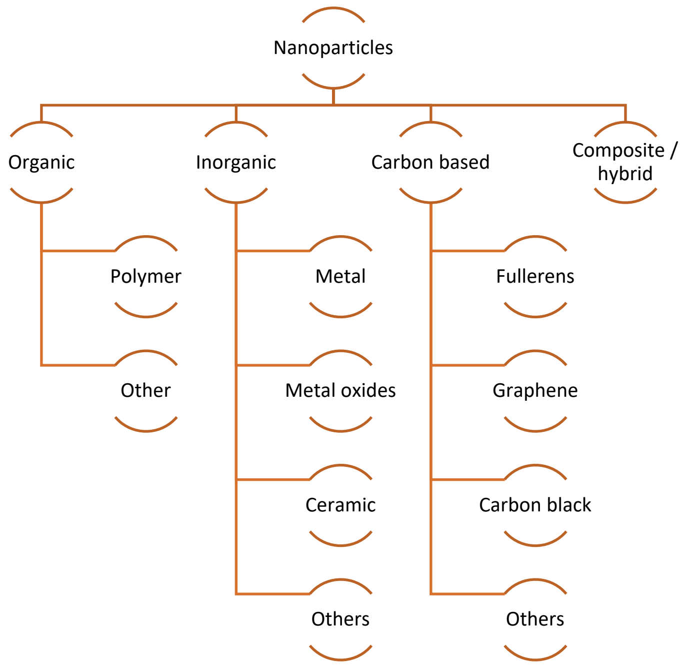


Figure 2. Nanoparticles are used as additives in construction materials.

Another important group is carbon-based nanoparticles. The implementation of such materials gives very good results; however, the main problem in this case is the high price. These nanoparticles significantly increase the mechanical properties of the material but not only. Recent research shows that composites with carbon nano-additives, including graphene, enhance the electrical conductivity of the material [40,41]. Because of this property and excellent mechanical performance, today, carbon nanofibers, especially carbon nanotubes, are widely applied in different advanced technologies, including the energy industry and the production of different types of sensors [42,43].

The last group is composite/hybrid nanoparticles. These materials joined one or more of the previously mentioned nanoparticles inside a group or between them. Thanks to this, they can influence different material properties by showing a synergistic effect [42,43]. The joining mechanism can work in different ways, including as an absorbing–desorbing mechanism, nanoencapsulation or covering one material by a nano-layer of another material [44–46]. The most popular applications for these materials are advanced technologies, including medical applications, where the combination of properties is necessary to obtain tailored properties of the material [47,48]. In the case of asphalt, this kind of mechanism was investigated with micro-additives to obtain the self-healing properties of the material [44].

3.2. Micro and Nano-Additives Dispersion

The distribution of the nanocomponents in the volume of the materials is connected with different parameters, including the particle's physical and chemical properties as well as the properties of the matrix [20,49]. For modelling purposes, it is assumed to be the best representation of existing processes in the material as colloid or aquatic environments [50,51]. In such an environment, the different processes are analyzed, including particle agglomeration, sedimentation, dissolution and chemical transformation [49,51]. Among these properties, the tendency to agglomeration has a crucial meaning for the further behavior of the particles. For the proper dispersion of particles, several methods are applied as potential prevention, including sonication using ultrasound [52,53].

Today, in asphalt and bitumen materials, this cumulative distribution is applied very rarely. Some research in this area was conducted just to test advanced properties [44,54]. One of the possibilities is to use hybrid nanoparticles with properties of phase-changing materials for application in the road area, which can help regulate the temperature in the city [54]. This solution seems to be especially attractive for roads and pavement in cities that influence air temperature in the urban canopy layer, including pedestrian thermal stress and adjacent building energy loads [54]. In other cases, where similar properties are required in the whole volume, it is not a desirable tendency. In the case of application in asphalt and bitumen, even distribution in the whole volume is the most frequently applied. The emerging agglomeration is usually connected with potential materials discontinuity, which negatively influences the mechanical properties. The most problematic area is the joint between the asphalt and aggregate and their adhesion [55,56].

Another problem area that is analyzed as a part of particle behavior is the manufacturing process: the shape of the particle can play an important role in flow dynamics during the production process and the mobility of the nanoparticles in the material [49,57]. It is also possible to steer the shape in the case of designing drug delivery systems or other solutions where the material can release additional substances [49,57]. From the processing point of view, the spherical (circular) particles should have a positive influence on the material's workability or viscosity. In the case of porous materials or microfibers, they can decrease the material's processing properties. Another nanocomponent has the tendency to create an internal layer in the volume of the material, especially in the case of films, layers, sheets, plates, etc.

Among the chemical properties, the important include the surface characteristic and internal structure. These features are also partly connected with physical properties and thermodynamic behavior [58–60]. Most important in this case is the coherence between a nanoparticle and matrix material and the interactions among the particles.

The improper physical and chemical properties of nanoparticles can cause some potential problems with obtaining the required materials properties. The basic problems connected with dispersion are outlined below [59,60]:

- Particle agglomeration in points, which locally changes the material's properties;
- Liner agglomeration that could cause the decoherence of material;
- Lack of cohesion particle—matrix and microvoids that weaken the material.

3.3. Area of Applications of Nano-Additives—Influence on Material Properties

The nanoparticles have various influences on prepared composites. These influences are dependent on the used particle, used matrix, particle treatment, amount of used nano-additives, and other factors. Overall, the research shows that the properly selected additives could significantly improve the thermal, mechanical, rheological, and barrier properties and conductivity [58–61], including anti-corrosion properties and wear resistance [31,62–67]. Moreover, they could significantly change the magnetoelectric properties [68,69]. They influence other special properties of selected materials, such as optical properties or biocompatibility [70,71]. The nanoparticles affect not only the final properties but in many cases also processing properties in traditional technologies as well as in modern ones, such as additive manufacturing [48,72–76]. They allow us to obtain better quality products most effectively.

Nowadays, nanocomposites find applications in many areas and industries. One of the most important is various protective coatings with nano-dispersed particles [65]. This coating has usually significantly increased operational characteristics, including corrosion resistance [65,67]. They can be applied to different materials, including steel and concrete [66,67]. Nanoparticles find similar applications in the packaging industry, where they are used as an antioxidant film. Other areas of application in this industry are high-performance packages with advanced electronic properties for packing valuable goods [48,73]. This kind of packing very often allows us to monitor the condition of the delivery [48].

The enhancement of mechanical, thermal, and electrical properties of nanocomposites is used also in multiple electronic applications [63,64]. One of the interesting examples is self-powered wearable electronic devices, including clothes. This kind of device allows for converting electrical energy from magnetic fields [68]. It is very interesting for the development of sustainable power sources/sensing tools [68] or for use for sensors for health monitoring. The nanomaterials are also widely applied in medicine, including polymer-based bone cement and drug delivery systems, where the possibility of encapsulation of substances inside nanoparticles is used [31,72].

The improvement of mechanical performance is used for structural applications, including additive manufacturing technology [69,76]. In particular, nanoparticles are applied to increase the parameters for filaments and enhance inter-layer bonding in extrusion techniques [74]. The nanoparticles are also used for filaments in electronic applications [75].

The important area of application for nanoparticles is optic properties, including the production of projection screens and passive and active micro-optical devices like beam splitters and a Pockels modulator [70,71]. The nanoparticles were also investigated for the mining industry as an improvement for drilling fluid [77].

The most important current applications of nano-additives are presented in Figure 3.

Figure 3. Selected application of nanoparticles and nanocomposites.

Among these examples, some of the properties of nano-additives enable them to be potentially used for application in asphalt and bitumen. The most obvious are connected with increasing the material properties and modification of the processing parameters. However, other modern applications can provide inspiration for creating new road products, including intelligent road surfaces that will be resistant to weather conditions and that can support navigation. Moreover, it has a completely new function, for example, air purification. These novel ideas do not require a completely new solution, but they use some already investigated products and adapt them for this specific purpose [78–80]. Today, this function seems to be a futuristic dream, but their application could be not so far in the future. In the last century, the plan for adding nano-components into asphalt and bitumen materials seemed to be unrealistic.

The idea for using nanomaterials as an additive for asphalt and bitumen materials was born in the XXI century [81,82], but the micro-additives started to be tested several years earlier. Both of these types of additives are beneficial, but there are some differences between them. The most important issue is that nanoparticles are more evenly distributed in asphalt compared to microparticles; it helps with forming network structures, hindering the propagation of internal microcracks [9]. It is also worth stressing that due to the large surface to volume ratio of nanoparticles, they have great potential for improving the rheological properties of asphalt and the adhesion of asphalt aggregates [83].

Despite the number of advantages mentioned above, the nanocomposite materials based on polymers have also some disadvantages [84]. The most important barrier to the wider application is price. Nanomaterials are relatively expensive modifiers, and their application is usually limited to advanced technologies where the price of the raw material is not a significant part of the final product. Other disadvantages are connected with the compatibility between nanoparticles and matrix and the proper dispersion of additives in the volume of material. It is also worth mentioning the health risks connected with the usage of nanoparticles. Some analyses show that they can be potentially harmful to humans, so safety procedures during manufacturing are required [2,4].

4. Types of Micro- and Nano-Additives Applied to Asphalt and Bitumen

Nowadays, different types of micro- and nano-additives are tested for use in asphalt and bitumen materials [1,85]. However, the majority of the research showed positive results. They were provided only on a laboratory scale, and their implementation requires additional research works on a larger scale [81,86]. The applied additives can be divided into some basic groups:

- Mineral-based nanomaterials: nano-silica, nano-clay, and nano-hydrotalcite.
- Oxide-based nanomaterials, including: titanium dioxide (TiO_2), alumina oxide (Al_2O_3), and zinc oxide (ZnO),
- Carbon-based nanomaterials, such as: carbon nanotubes (CNTs) or graphene oxide (GO).
- Others, for example nano-cellulose.

This classification is slightly different from those presented before in Figure 2. However, it also involves all the most important particle groups. Moreover, it seems to be a better fit with the properties of asphalts and bitumen, and because of that, it was selected for this section.

4.1. Mineral-Based Micro- and Nano-Additives

The most popular type of micro and nano-additives seems to be mineral-based materials, such as nano-silica and nano-clay [9,15,87,88]. This group of additives usually enhances the physical and rheological properties as well as the durability of asphalt mixtures [87,89–91]. This group also improves rutting resistance, fatigue properties and temperature susceptibility [92]. Some authors also argue for the positive environmental impact of these mixtures but without supporting this claim by wider analysis [82,87,89,90]. The provided research also shows that this kind of additive works better in higher temperatures and improves the high-temperature performance of materials, but it could have a slightly negative effect on cracks caused by low temperature [89,90,92]. The most promising results were obtained with nano-silica and nano-clays.

Sodium montmorillonite is another possible addition to bituminous binders in this group. This material has a layered aluminosilicate (clay) with a tactoid structure, which can be converted into nanosized particles (nano-clay) in two ways: by intercalating continuous medium molecules into the interlayer space of the clay or by exfoliating the clay layers from each other [7,93]. As a raw material, this clay has hydrophilic properties and because of that, it creates agglomerations in the material. For a successful application, it is required to change the properties of these additives to hydrophilic ones [93]. It causes the clay to exfoliate into nanosized particles in bitumen and improves the properties of the whole mixture. It is worth noticing that a similar improvement of properties by changing the surface from hydrophilic to hydrophobic was shown by the same authors for nano-silica [94].

Among the mineral additives, one of them with increasing popularity and the number of investigations is halloysite [95,96]. Halloysite is an aluminosilicate clay mineral with the empirical formula $Al_2Si_2O_5(OH)_4$. The application of this material is connected with the improvement of the properties of materials, including flame retardancy [95], electrostatic properties [96], and others. The exemplary SEM images for halloysite nanomaterial are presented in Figure 4.

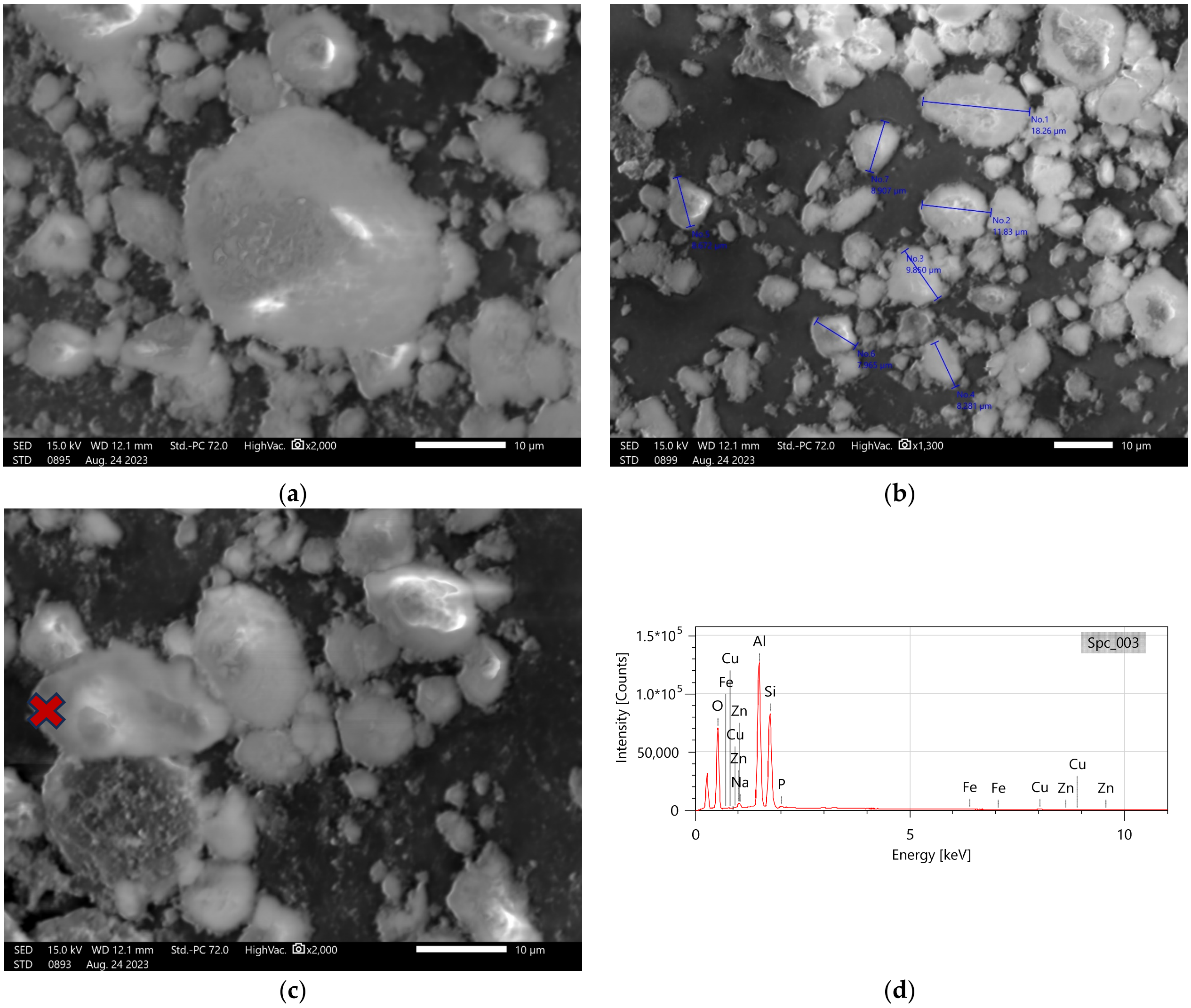


Figure 4. The morphology of halloysite: (**a**) SEM image of halloysite in magnification 2000×; (**b**) SEM picture with measured and marked selected particle dimensions; (**c**) SEM picture with marked point for EDS analysis; (**d**) results of EDS analysis for halloysite.

The structure of the material is typical for mineral additives obtained in the mining process through crushing, grinding and milling. The shape of the grains is irregular with some sharp edges. In the case of the investigated material, the particles of the halloysite have an average dimension of about 12 μ; however, it is technically possible to obtain more fine materials in the milling process. The structure of the needle-like crystals for this material can be revealed using transmission electron microscopy (TEM) [97]—see Figure 5.

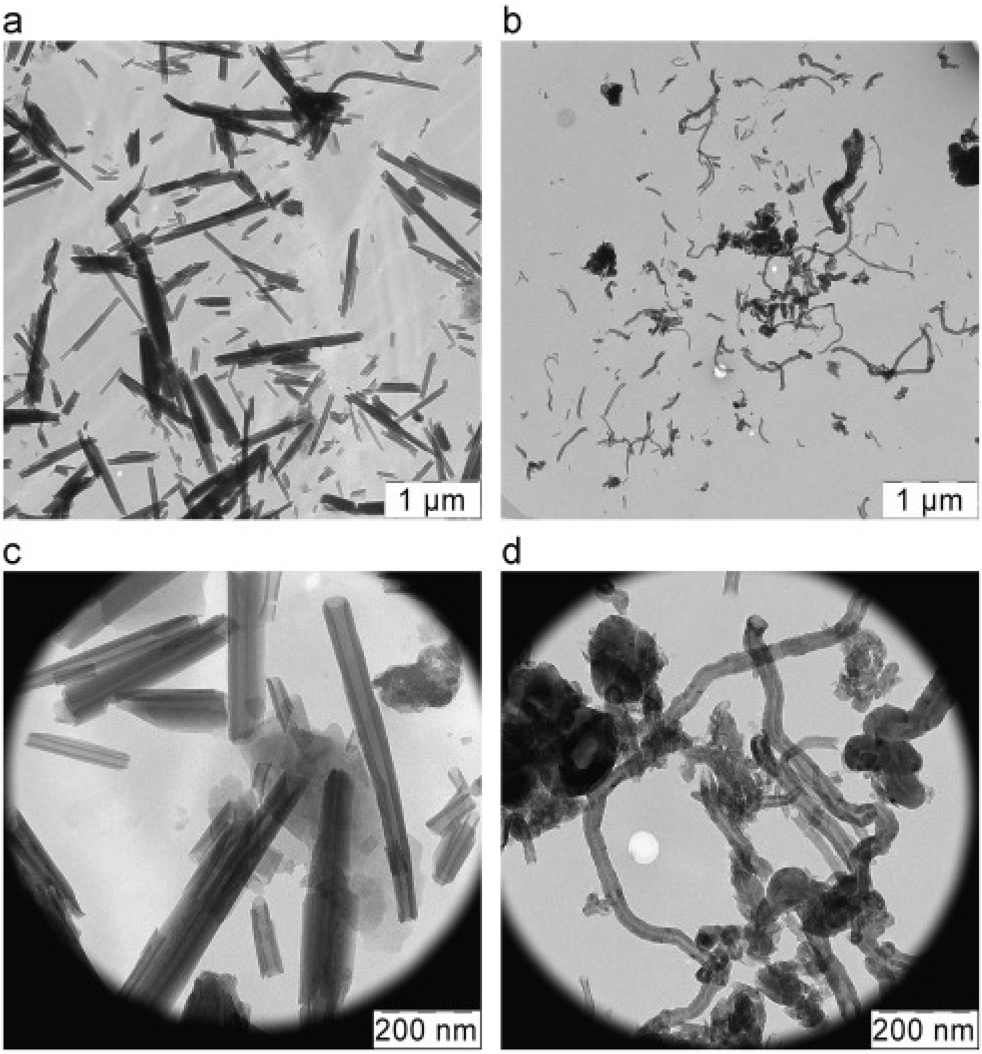


Figure 5. TEM images of halloysite (**a**,**c**) and carbon nanotubes (**b**,**d**) were used in the experiments [97].

Additionally, EDS analysis was provided for the selected point (Figure 4 and Table 1).

Table 1. Elemental and oxide composition obtained from EDS investigation.

Elemental Composition		Oxide Composition	
Chemical Formula	**% Mass**	**Chemical Formula**	**% Mass**
O	38.01 ± 0.09	Na_2O	0.49 ± 0.01
Na	0.41 ± 0.01	Al_2O_3	47.12 ± 0.10
Al	29.46 ± 0.06	SiO_2	46.74 ± 0.12
Si	27.10 ± 0.07	P_2O_5	1.15 ± 0.02
P	0.64 ± 0.01	FeO	0.51 ± 0.02
Fe	0.48 ± 0.02	CuO	2.18 ± 0.07
Cu	2.11 ± 0.06	ZnO	1.81 ± 0.07
Zn	1.77 ± 0.07		

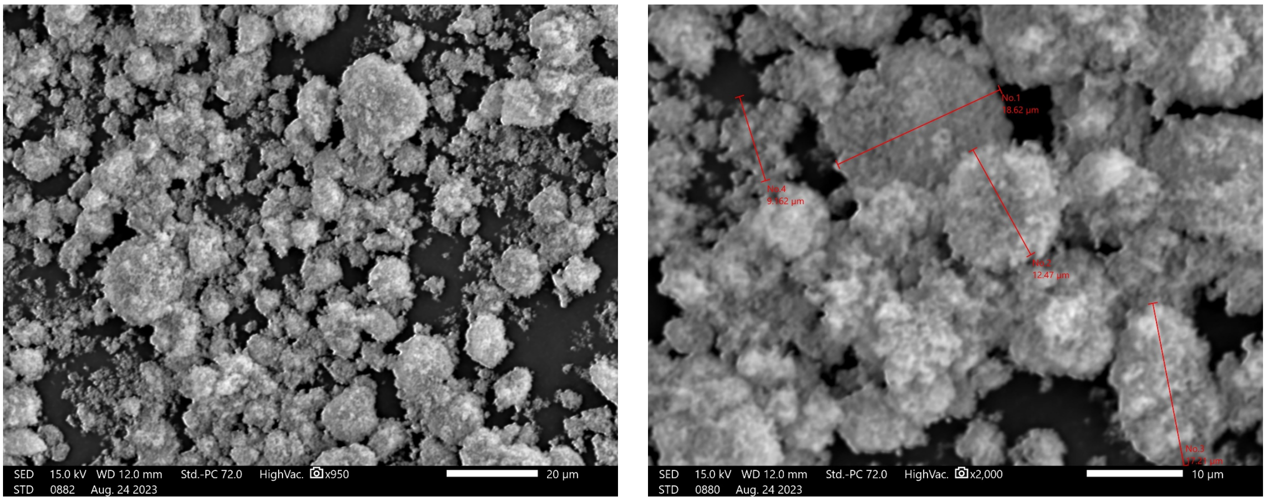
EDS confirms the basic elements that are characteristic of this mineral, such as Si and Al, but also shows some additional elements. Their presence is typical for materials obtained from the mining process.

4.2. Metal and Oxide-Based Micro- and Nano-Additives

Another group of nano-additives that was widely investigated is oxide-based nanomaterials, especially titanium dioxide (TiO_2), alumina oxide (Al_2O_3), and zinc oxide (ZnO) [9,15,98–100]. The other additives have been studied to a small extent, such as nano-cuprum dioxide (Cu_2O) [101]. These types of additives efficiently enhance the elastic recovery and increase the asphalt stiffness and rutting resistance of asphalt at high temperatures [89,90,102]. Other research studies also show the improvements in the physical performance and aging resistance of asphalt and bitumen [103,104], including a decreased creep stiffness and improved softening point and rutting factor of the asphalt binder [105]. The research shows that the usage of this kind of additive helps in obtaining asphalt mixtures with greater high-temperature stability, low-temperature cracking resistance and lower moisture susceptibility [17,106]. Another advantage of this kind of additive is the increased stiffness of asphalt, which could be beneficial in reducing the permanent deformation of the pavement [107,108]. Additionally, particles from this group such as nano-ZnO and nano-TiO_2 are semiconductors and absorb ultraviolet (UV) rays or scatter them [106,109,110]. However, UV light has adverse impacts on asphalt [110,111]. The research shows that the most advantageous influence is generated by the materials with layered characteristics, which remarkably enhance the resistance of asphalt material to UV aging and thermo-oxidative aging [112,113].

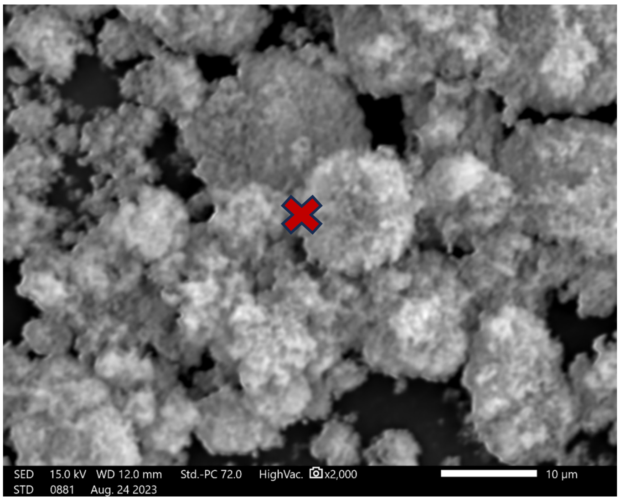
Among these materials, one of the most promising seems to be ZnO. It is considered an important additive for pavement construction materials from both a technical (as an asphalt binder modifier) and an environmental point of view [114]. The latest publications show that it can play an important role in helping to improve urban air quality [114–116]. It is possible because nano-ZnO particles have a large surface area relative to their size and high catalytic activity. They have irregular shapes that are related to a method of production (Figure 6).

The analyzed particles of the material have a dimension of about 12 µm with a well-developed surface area. This area is a consequence of the used production method for ZnO nanoparticles. Additionally, EDS analysis was provided for the selected point (Figure 6 and Table 2).

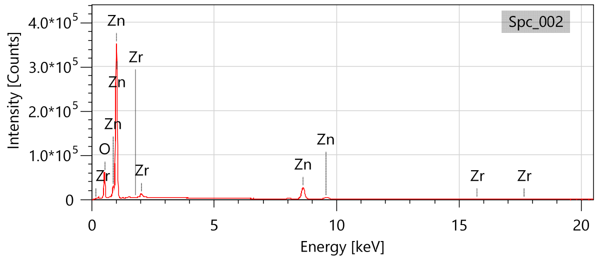


(**a**) (**b**)

Figure 6. *Cont.*



(**c**)



(**d**)

Figure 6. The morphology of nano zinc oxide: (**a**) SEM image of nano zinc oxide at 950× magnification; (**b**) SEM picture with measured and marked selected particle dimensions; (**c**) SEM picture with marked point for EDS analysis; (**d**) Results of EDS analysis for nano zinc oxide.

Table 2. Elemental and oxide composition obtained from EDS investigation.

Elemental Composition		**Oxide Composition**	
Chemical Formula	**% Mass**	**Chemical Formula**	**% Mass**
O	13.80 ± 0.04	ZnO	95.59 ± 0.31
Zn	82.62 ± 0.26	ZrO_2	4.41 ± 0.03
Zr	3.58 ± 0.03		

EDS shows that the analyzed sample is mainly ZnO with some amount of zirconium oxide (ZrO_2).

4.3. Carbon-Based Micro- and Nano-Additives

Another interesting group of nano-additives consists of carbon-based nanomaterials, including nanotubes and graphene oxide [117,118]. The implementation of such materials gives very good results; however, the main problem in this case is price [117,119]. Multi-walled carbon nanotubes (MWCNTs) contain nested single-wall carbon nanotubes in a nested, tube-in-tube structure. The structure of a single nanotube is not well visible under an SEM microscope. The material seems to be some agglomeration of nanoparticles (Figure 7).

In this case, some different structures made by the materials were detected. The particles of the material, or rather its agglomeration, have an average dimension of about 15 μm (Figure 6). The structure of the single tube for this material can be revealed using TEM [97]—see Figure 5. Additionally, EDS analysis was provided for the selected point (Figure 6 and Table 3).

According to expectations, the material is composed mainly of pure carbon. The oxide analysis also does not detect carbon oxides. It is worth mentioning that in this case, carbon was left as the main expected element of the composition; however, the amount cannot be treated as reliable for the applied method of analysis because a carbon pod was used for sample preparation. The small amount of cobalt oxide is an effect of the manufacturing process for MWCNTs.

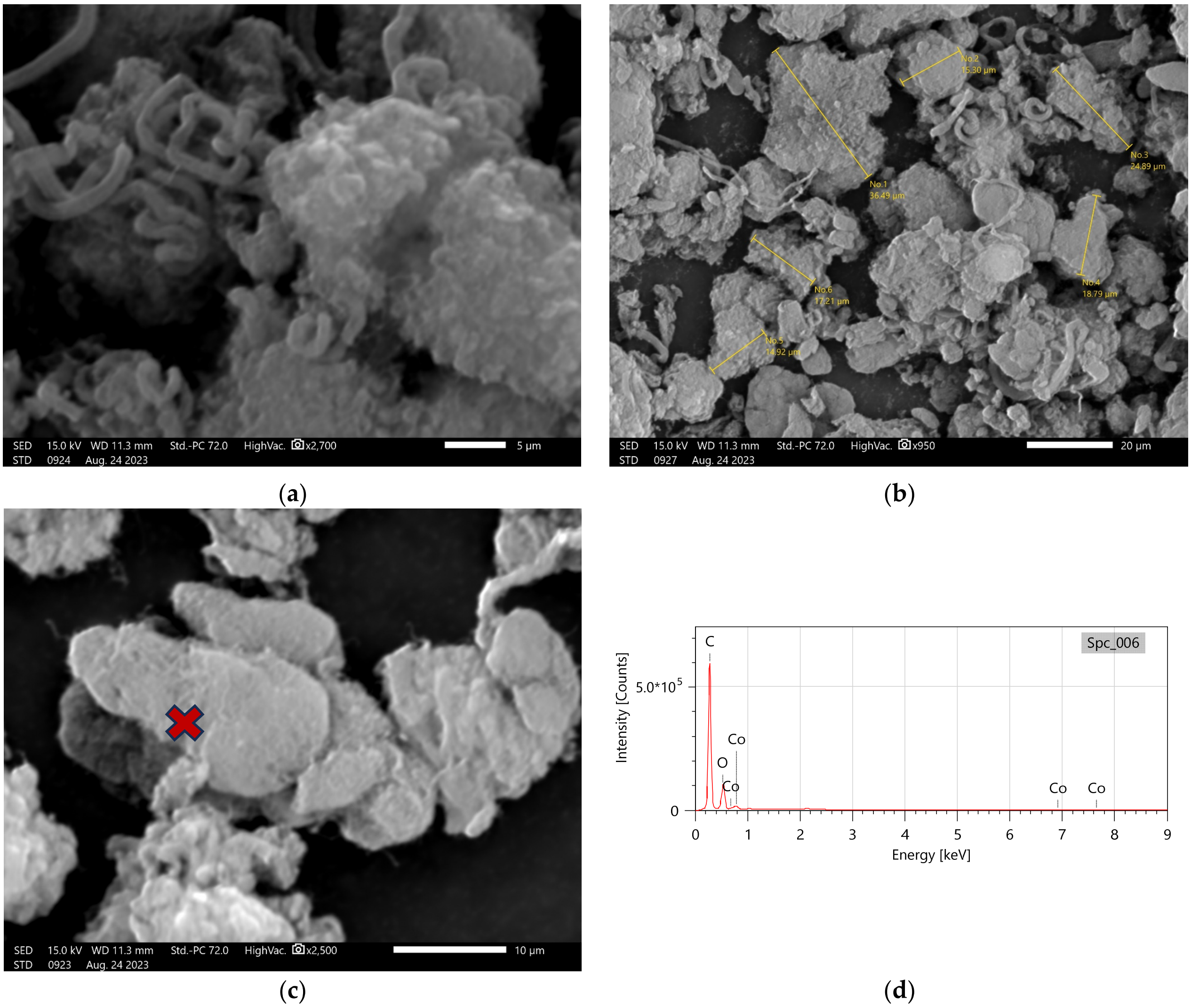


Figure 7. The morphology of carbon nanotubes: (**a**) SEM image of carbon nanotubes at a magnification of 2700×; (**b**) SEM picture with measured and marked selected particle dimensions; (**c**) SEM picture with marked of point for EDS analysis; (**d**) results of EDS analysis for carbon nanotubes.

Table 3. Elemental and oxide composition obtained from EDS investigation.

Elemental Composition		Oxide Composition	
Chemical Formula	**% Mass**	**Chemical Formula**	**% Mass**
C	72.47 ± 0.03	C	99.08 ± 0.05
O	27.09 ± 0.06	CoO	0.92 ± 0.02
Co	0.44 ± 0.01		

4.4. Other Micro- and Nano-Additives

In contemporary research where micro- and nano-additives were applied into asphalt and bitumen materials, organic nano-additives such as nanocellulose are playing an increasingly important role [16,93]. Nanocellulose can be used for the manufacturing of nanocomposite bitumen binders, which creates a microfibrillar network in the bitumen and increases its cohesive strength and resistance to rutting [16]. Nanocellulose is nanostructured cellulose, which is an organic material (Figure 8). It could have different shapes; in the case of the observed material, the shape was spherical.

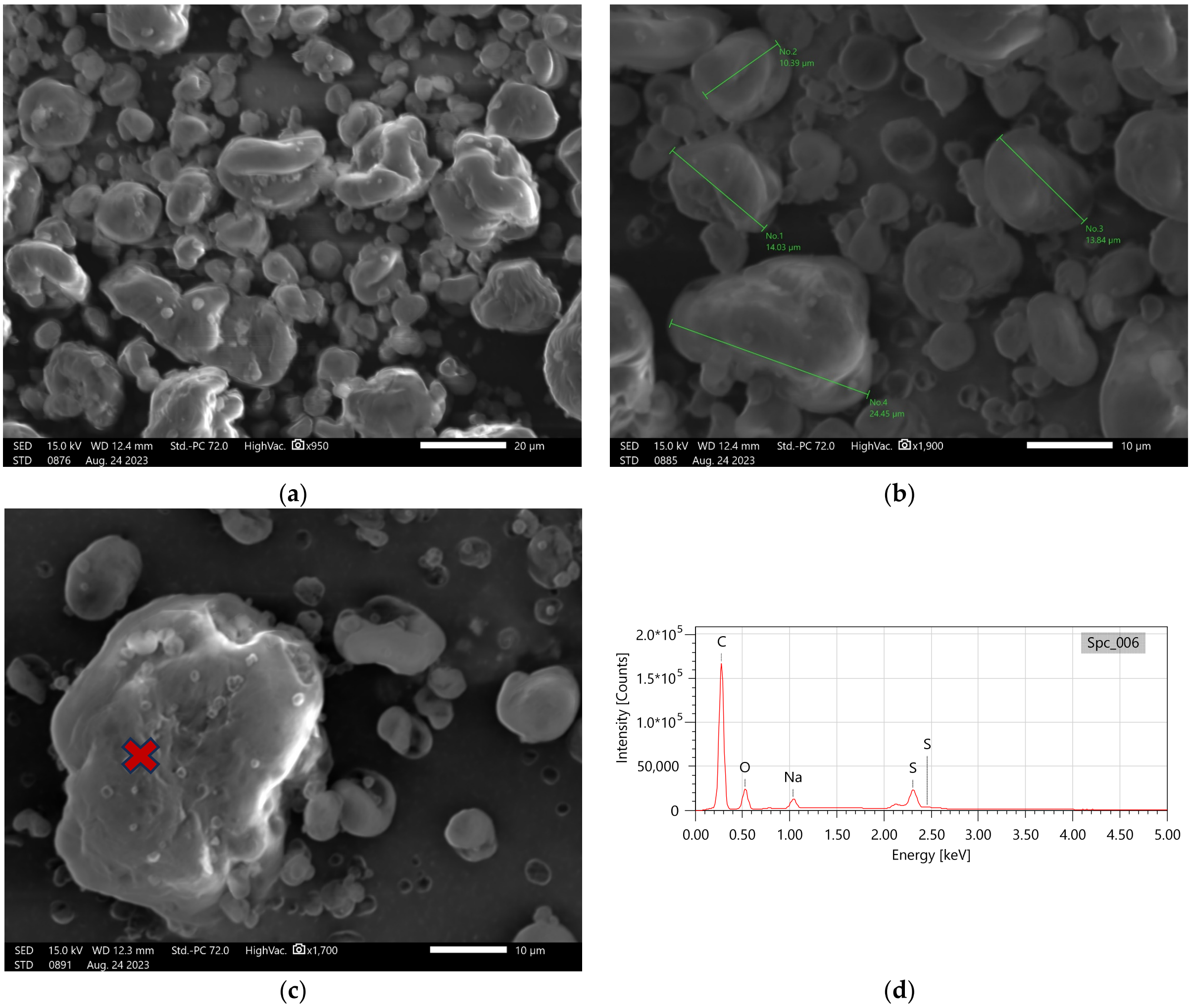


Figure 8. The morphology of nanocellulose: (**a**) SEM image of nanocellulose at a magnification of 950x; (**b**) SEM picture with measured and marked selected particle dimensions; (**c**) SEM picture with marked of point for EDS analysis; (**d**) results of EDS analysis for nanocellulose.

These particles had a regular shape that is close to spherical with a particle dimension of about 15 µm (Figure 8). Additionally, EDS analysis was provided for the selected point (Figure 8 and Table 4).

Table 4. Elemental and oxide composition obtained from EDS investigation.

Elemental Composition		Oxide Composition	
Chemical Formula	**% Mass**	**Chemical Formula**	**% Mass**
C	74.74 ± 0.07	C	85.18 ± 0.08
O	19.02 ± 0.08	Na_2O	2.99 ± 0.02
Na	2.06 ± 0.02	SO_3	11.83 ± 0.05
S	4.18 ± 0.02		

The material should be composed of carbon, which is in line with the expected composition of organic particles. However, the analysis shows also some amount of sodium oxide and sulfur oxide. Their presence is most likely the a product of the production process. Because on an industrial scale, cellulose is obtained from wood using the sulfite or natron method, which involves separating (chemically decomposing) lignin from cellulose [120]. Similarly to the previous case, the carbon was left, because this element was expected to be one of the most important in the composition of organic particles. However, their amount cannot be treated as reliable for the applied method of analysis, because a carbon pod was used for sample preparation, and the amount of carbon is probably higher than in reality. So, the analysis does not provide proper quantitative results.

5. Implementation of Micro- and Nano-Additives into Asphalt and Bitumen

The information about the most important research in this area is summarized in Table 5.

Table 5. Nano-additives applied to bitumen and asphalts.

Additive	Matrix Material	Influence/Main Findings	Source
Nanoscale tire rubber	Modified asphalt pavement	• Nanocomponents stabilized the molecular weight distribution and decreased the oxidative condensation reaction of the asphalt matrix during weathering. • Nanoparticles inhibit the weathering reaction including asphalt condensation and asphalt oxidation. • Modified asphalt had more stable crack resistance at low temperatures and stable deformation resistance. Moreover, it has excellent elasticity at high temperatures during weathering.	[121]
Nanocellulose (1% aqueous dispersion of microfibrillated cellulose)	Pickering bitumen emulsion	Production of bitumen emulsions using cellulose and the subsequent drying of the results. Pickering emulsions can be an alternative and practical way to produce nanocomposite bitumen binders with outstanding properties	[16]
Hydrophobic clay (montmorillonite nanoparticles (10–30%))	Bitumen binder	• Hydrophobic clay exfoliates to nanosized particles in bitumen unlike hydrophilic ones. • Hydrophobic clay causes bitumen gelling with increasing stiffness and yield stress. • Hydrophobic clay more effectively increases the bitumen cohesion and adhesion.	[16,93]
Nano-TiO_2/ZnO (and additionally basalt fiber)	Asphalt	• Rheological properties of basalt fiber and nano-TiO_2/ZnO composite improve the performances of asphalt binder. • Basalt fiber and nano-TiO_2/ZnO composite can delay the asphalt binder aging process. • The optimal content of nano-TiO_2/ZnO is 4% in modified asphalt while the content of basalt fiber is 6%. • FTIR results suggest that there was no chemical reaction between basalt fiber, nano-TiO_2/ZnO, and asphalt, and the modification mechanism is mainly of a physical nature.	[9]
nano-$CaCO_3$	Stone mastic asphalt (SMA)	• Nano-$CaCO_3$ increased the fatigue life and rutting resistance of the SMA mixture. • Moisture damage resistance of the SMA mixture was ameliorated by adding nano-$CaCO_3$. • Almost in all cases, the 0.9% nano-$CaCO_3$-modified SMA mixture showed the best behavior.	[83]
Carbon nanotubes (CNTs) (0.1%, 0.5%, and 1% by mass of asphalt cement)	Asphalt	• The results exhibited that modifying asphalt cement with CNTs decreased its penetration and increased its kinematic viscosity and softening point. • The Marshall stability increased with CNTs but there was no significant difference at 0.5 and 1.0 wt%, while Marshall flow decreased with CNTs. • The results of the wheel tracking test showed that the rut depth decreased by 45% upon adding 0.5% CNTs by weight of asphalt cement; also, this percentage of CNTs led to an improvement in low-temperature cracking and the indirect tensile strength of the asphalt concrete. • The additive of CNTs into asphalt cement enhances the performance of asphalt concrete pavement in both hot and cold weather, which in turn prolongs the pavement's service life and reduces the maintenance expenses.	[119]

Table 5. *Cont.*

Additive	Matrix Material	Influence/Main Findings	Source
Graphene platelets (GnPs), 0.5%, 1.0%, and 1.5% by weight of asphalt content	Asphalt	Graphene platelets enhance the mechanical properties of asphalt mixture and its performance.	[18]
Nano-hydrotalcite	SMA	• The modification with nano-hydrotalcite induced smaller evolution in the fatigue resistance parameters, indicating enhanced aging resistance. • Regarding surface characteristics, the modified nano-additive asphalt mixture presented approximately similar behavior to the control materials, having higher skid resistance and lower mean texture depth.	[122]
Graphene oxide (GO)	Asphalt	• SBS-modified asphalt showed better viscoelastic properties via 0.3 wt% graphene oxide addition. • Internal micro-state structures of modifier and base asphalt were enhanced.	[118]
Nano-clay, nano-lime, and nano-alumina (1, 2.5, and 4%)	Cold recycled pavements	Nano-clay, nano-lime, and nano-alumina increased the resilient modulus and fatigue life of cold recycled samples.	[123]
Nano-clay ratio of 6%	Asphalt binder	Utilizing nano-clay and SBR enhanced the rutting resistance of asphalt and HMA mixtures.	[124]
Nano-SiO_2, nano-zero-valent iron, and nano-bentonite	Asphalt mixtures	Nano-SiO_2, nano-zero-valent iron, and nano-bentonite ameliorated the rutting behavior, moisture damage resistance, and fatigue performance of asphalt mixtures.	[125]
Nano-clay and Nano-iron	AC 14 mixture	Results indicated that the moisture damage resistance of AC 14 mixtures was enhanced by nano-iron. Moreover, the performance of the AC 14 mixture against aging was improved by nano-clay.	[5]
3% of nano-clay	HMA mixtures	Utilizing nano-clay improved the fatigue behavior of the HMA mixture.	[126]
Nano SiO_2 and Nano TiO_2 (0.3, 0.6, 0.9, and 1.2%)	SMA mixtures	• The addition of nanomaterials can improve the mechanical behavior of SMA mixtures. • Nano-SiO_2 and TiO_2 increased the fatigue life and decreased the rut depth of the SMA samples.	[127]
Silica nanopowder (0.1, 0.3, and 0.5%)	HMA mixtures	• The aged modified asphalt samples with a nano-silica ratio of 0.3% had better rutting behavior. • Moisture resistance of HMA mixtures modified with a nano-silica ratio of 0.3% is higher than other modified samples and control samples. • Energy saving is improved by modification.	[128]
Carbon nanotubes (CNT) 0.005% of bitumen weight	Bitumen	Modifying bitumen by CNT changes the binder properties and improves the properties of the asphalt.	[117]
Nano-TiO_2	Asphalt	• Replacing 5% of the bitumen by nano-TiO_2 improves the creep behavior of the asphalt mixtures. • The addition of nano-TiO_2 can improve the creep behavior of asphalt mixture even at high temperatures and prevents tensile cracks from being easily generated by horizontal tensile stresses.	[129]
Nano-zinc oxide (ZnO)—1, 3, 5, and 7%	HMA	Nano-ZnO raised the fatigue cracking resistance of HMAs.	[130]
4% of Nano-TiO2	HMA mixtures	Nano-TiO_2 improved the resistance to permanent deformation.	[131]

Table 5. *Cont.*

Additive	Matrix Material	Influence/Main Findings	Source
5% of Nano-SiO2 powder	SMA mixtures	• Nano-SiO_2 and SBS improved the ITS values of mixtures. • Nano-SiO_2-modified samples were more resistant to moisture damage compared to modified samples with SBS. • Nano-SiO_2 and SBS raised the stiffness modulus of mixtures.	[132]
1% of nano-powdered rubber VP401 and 1% of VP501	HMA mixtures	The rutting and water stability of mixtures experienced improvements by adding VP401 and VP501.	[133]
0%, 0.3%, 0.65%, 1%, 1.5%, 2.5%, 5%, and 7% of graphene	Asphalt binders	• Graphene had good dispersion at dosages below 1.5% and good compatibility. • Graphene enhances the rutting performance.	[134]
Graphene nanoplates	Polymer-modified asphalt concretes	Graphene pellets could significantly enhance mechanical performances.	[135]
5 to 15 wt% of hydrophilic or hydrophobic nano-silica	Bitumen BN 90/10	• Nano-silica enhances the elasticity of bitumen and gives it yield stress behavior. • A large amount of nano-silica improves bitumen strength independent of its surface. • Hydrophilic silica decreases bitumen adhesion, but the hydrophobic one improves it.	[94]

In recent years, it has become more and more popular to join different nanomaterials with other additives, such as fibers, rubbers, or styrene–butadiene–styrene block copolymer [9,15,81] as well as some micro-additives [98,100,136]. It allows avoiding some negative effects and obtaining so-called synergy effects between different kinds of additives [103,137,138].

Despite the large number of advantages, the nanomaterials also have some disadvantages. The most important barrier to the wider application is price. Nanomaterials are relatively expensive modifiers, and their excessive usage for modifying asphalt mixtures is not economical [83,85]. Other disadvantages are related to the compatibility between nanomaterial modifiers and asphalt. The dispersion of nanomaterial in asphalt is a critical challenge for the application of nanomaterials in improving the aging resistance of asphalt [85]. It is also worth mentioning the health risks connected with the usage of nanoparticles. Some analyses show that they can be potentially harmful to humans, so safety procedures during manufacturing are required [2]. It is also worth mentioning that all material modification can significantly affect the environment. The material in asphalt and bituminous materials has a significant impact on the estimation of environmental burden [139].

6. Conclusions

The development of nanocomposites is an important trend in modern material science with the researching providing strong practical applications in this area. These nanocomposites can improve the materials' properties for the most advanced applications as well as provide new properties to obtain multifunctional materials for the modern economy. It seems to be one of the important research areas for many industries, including asphalt and bitumen applications. The following conclusions can be formulated based on the presented review:

- The development of asphalt and bitumen using micro- and nano-dispersion additives is an important trend in modern material science and civil engineering. It seems to be one of the important research areas for an economy that will have a wider context for the improvement of infrastructure, including transportation systems.
- A wide range of micro- and nano-additives have been tested in asphalt and bitumen materials, including mineral, metal and organic particles.

- Mineral particles very often require surface modification to achieve good adhesion with asphalt and bitumen matrix because of their hydrophobic character.
- The micro- and nano-additives could improve the properties of asphalt and bitumen mixtures as well as influence the production process, including the reduction in temperature.
- The most commonly modified properties are fatigue and deformation resistance.
- It is worth noticing that one of the barriers to wider applications is the lack of international standards, including a lack of regulation connected with specific problem micro- and nano-dispersion additives.
- Other existing challenges include the safe use of nanomaterials, long-term properties, materials' durability, and the proper dispersion of nano-additives for asphalt and bitumen.

These issues are important research perspectives that to date have received little attention in the world literature.

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