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REVIEW

of **M.Sc. Jyoti Rashmi Nayak's** dissertation entitled

"Analysis of impact of selected natural waste fibres and ashes on properties of mortars"

1. Formal framework of the review

The formal framework for the preparation of this opinion is the decision letter of the Chairman of the Council of the Discipline Civil Engineering Geodesy and Transport of the Silesian University of Technology, Mr Marcin Staniek, D.Sc., Professor of Silesian University of Technology, dated 19.01.2024, containing an assignment to perform this review based on the resolution of 21.12.2023.

The substantive basis is the text of the doctoral dissertation by Ms Jyoti Rashmi Nayak entitled "Analysis of impact of selected natural waste fibres and ashes on properties of mortars", attached to the order.

The legal basis for the review is the Act of 20 July 2018. Law on Higher Education and Science (Journal of Laws 2018, item 1668).

2. Subject of the review

The subject of this review is a doctoral dissertation by Ms Jyoti Rashmi Nayak, M.Sc., entitled. "Analysis of impact of selected natural waste fibres and ashes on properties of mortars". The dissertation's supervisor is Jerzy Bochen, D.Sc., Professor of Silesian University of Technology and the associate supervisor is Małgorzata Gołaszewska, D.Sc. The dissertation was prepared in the Department of Building Process and Building Physics at the Faculty of Civil Engineering at the Silesian University of Technology in Gliwice.

3. Formal characteristics of the dissertation

The dissertation concerns the possibility of utilising two types of natural organic waste in cement and cement-lime mortars. The first was natural jute and sisal fibres acting as reinforcement for the analysed

composites in the form of dispersed reinforcement. The second type of materials were additives with expected pozzolanic properties to the cement binder resulting from the combustion of rice husks and sugarcane bagasse.

The dissertation has been written in English and edited into a total of 155 A4 pages. It contains a total of 11 chapters preceded by an abstract, a table of contents and an explanation of the abbreviations and symbols used. The work is finished with a list of publications used, as well as a list of tables and figures. Also included are two appendices with graphs showing the compressive strength results obtained for all combinations of mortar compositions containing both mineral additives and organic fibres.

In the first chapter, which serves as an introduction to the main part of the literature analysis, the Author outlines the current general problems associated with sustainable development in building constructions, i.e. among other things, CO2 emissions resulting from cement and steel production. She also draws attention to the depletion of natural resources and the still insufficient use of secondary raw materials. The PhD candidate points out that the use of both natural fibres and plant-derived ashes in cement composites can not only reduce the amount of CO2 emitted into the atmosphere as a result of reduced clinker use but also its partial sequestration during the plant growing season. An added value of the dissertation is the possibility of production of the title eco-friendly cement mortars with the above-mentioned additives with enhanced mechanical properties.

The second chapter is divided into two parts and is mainly based on the analysis of European standards for masonry and plaster mortars. In the first part, the Author presented the directions of applications of this type of cementitious composites in construction, their roles, classification and technological, physical and mechanical properties relevant from the application point of view. In conclusion, the Author draws attention to the possibility of modifying selected properties of the materials in question by using natural fibres and plant-derived ashes. The second part contains basic information on comparing the properties of cement and cement-lime mortars, including the roles of the individual components and their typical proportions.

The third part of the work is a fundamental analysis of the literature. Reports on the influence of various types of additives to cementitious mortars on their selected properties. Although this chapter has been divided into 4 subchapters, it de facto consists of two parts, in which the Author analyses current reports on the properties of composites containing dispersed reinforcement in the form of natural fibres, but also as a kind of reference reinforcement - polypropylene ones. In this part, one can find a richly documented presentation not only of the properties of the fibres themselves but also of their chemical composition, significantly influencing their cooperation with the cement matrix, as well as their influence on the technological, physical and mechanical properties of mortars and concretes. In the second part, the literature review deals with the possibility of using secondary raw materials as cement supplements mainly in mortars. The scope of characterisation of raw materials includes mainly ashes resulting from the combustion of waste of plant origins, but also against the background of mineral additives generally known in concrete technology such as fly ash,

metakaolinite or blast furnace slag. The Author of the dissertation focuses her attention on the properties, analogous to those of fibres, of the technological physical and mechanical properties of cementitious composites.

Chapter 4, entitled "Aims and scope of the thesis", summarises the literature review and the resulting research objectives. On this basis, one thesis of the thesis was formulated as the starting point for the presented outline of the research programme. It is divided into two independent parts concerning the evaluation of the properties of mortars containing dispersed reinforcement of plant origin and ashes from the combustion of rice husks and sugarcane bagasse.

In Chapters 5 - 7, the PhD student characterises the materials used for the production of the analysed mortars and, referring to the relevant standards, describes the test methods used in the assessment of the properties of eco-friendly cement and cement-lime mortars. In addition to basic tests of the characteristics of the mixtures and hardened materials, i.e. consistency, aeration, drying shrinkage and mechanical properties, she uses more advanced instrumental methods to determine changes at the microstructural level of the composites, i.e. SEM scanning electron microscopy and MIP mercury intrusion porosimetry. The last part of the scope of the dissertation in question is a chronological run of the research stages and tables containing the compositions of the mortar variants that were the subject of the PhD student's research.

The eighth and ninth chapters constitute the main part of the dissertation, in which the author presents the results of research on mortars containing dispersed reinforcement made of natural, i.e. jute and sisal fibres and artificial fibres - polypropylene. The presentation of research results includes all the elements indicated earlier in the planned research programme. In the case of the description of the results of the application of rice husk ash and sugarcane bagasse ash as cement supplements in mortars, the configuration of the presentation of the results obtained is analogous.

In Chapter 10, the Author has included 19 mainly detailed conclusions concerning the course of the entire research programme carried out. The last two conclusions of a more general nature relate to the thesis set out in the dissertation. The last, eleventh, part of the dissertation deals with relevant research planned for the development of this type of composites in the future.

The dissertation contains 21 tables and 30 figures, which certainly required the PhD student to devote considerable effort to their preparation.

I find that the layout of the entire dissertation, the order of the results presented in Chapters 8 and 9, and the way they are presented are logical and clear. Furthermore, the selection of literature items is sufficient and well-suited to the topic of the dissertation.

4. Substantive evaluation of the dissertation

4.1. Evaluating the selection of the dissertation topic, the definition of its purpose and the formulation of the thesis

The title of the dissertation, 'Analysis of impact of selected natural waste fibres and ashes on properties of mortars', reflects its content well, although the work is more concerned with selected properties of these composites, i.e. basic technological, mechanical properties extended by microstructure analysis. The topic of the dissertation fits perfectly into the trend of sustainable development of construction at the level of manufacturing pro-ecological building materials. It should be emphasised that the Author's concept of using ashes from the combustion of plant-derived materials in cement mortars can have a very beneficial effect of limited CO2 emissions due to their similar level to the amount sequestered during plant vegetation. In addition, the direct use of natural jute and sisal fibres will have a positive effect on the CO2 balance in the overall calculation of the production of such eco-friendly mortars. In my opinion, the dissertation lacked a quantitative account of this issue, despite the already extensive literature in this field. I believe that during the defence of the thesis, the PhD candidate should discuss this issue more extensively.

The dissertation is based on a single thesis in which the Author assumes that both the presence of natural fibres and plant-derived ashes affect the physical, mechanical and microstructural properties of cement mortars. This seems quite obvious and does not require proof, especially as the Author does not define in what way - that is, whether it improves or worsens these properties. In the second part of the thesis, the Author assumes that, at a certain optimum level of fibre and ash content, it is possible to achieve a beneficial effect on the selected properties. In this case, it seems that it would be necessary to specify the properties in which the beneficial effect of the presence of the analysed additives is expected.

4.2. Assessment of the PhD Candidate's general knowledge of the discipline

The range of literature directly related to the use of ashes and plant-derived dispersed reinforcement in cementitious composites, which the Doctoral Student has studied and analysed, is remarkable, as it consists of 181 items. More than 27% of the cited papers come from the last 5 years, and more than half, i.e. 52%, from the last 10 years, which also confirms the recent interest of many research centres in this subject and proves the topicality of the research problem constituting the subject of this dissertation.

The Author describes the current state of knowledge in an orderly and clear manner, correctly using nomenclature specific to cementitious composites technology. The content of the individual chapters does not raise any major objections.

In my opinion, there is a certain lack of discussion on the effectiveness of plant-derived dispersed reinforcement. The way in which the composites are damaged and the work of fibres after the matrix has been

cracked is crucial for this type of composite, especially in the situation of loss of moisture content and therefore probable shrinkage of the fibres and consequent reduction of their adhesion to the matrix. This problem is discussed in the literature.

The dissertation also does not address the problem of biological corrosion, which may be the main cause of limiting the durability of this type of composite, but it should be noted that the Author is aware of this, because, as she states in Chapter 11, "Future work", she plans to develop issues with durability in general.

With regard to the additives supplementing the cementitious binder, in both cases of RHA (Rice Husk Ash) and SCBA (SugarCane Bagasse Ash), pozzolanic properties can be expected, according to the literature. This basic property strictly, has not been discussed despite the wide range of possibilities to verify this characteristic, for example by mechanical tests (determination of the SAI coefficient) or chemical tests, e.g. using the Frattini or Chapelle method.

Certainly, an important feature of building materials containing plant-based additives will be the resistance of future products to high temperatures under fire conditions. In my opinion, this problem should not be omitted from the dissertation either.

The above criticisms do not affect a positive assessment of the extent of the Doctoral Student's knowledge in the discipline, but may serve as a pretext for a broader discussion of these issues during the defence.

4.3.Assessment of the ability to conduct scientific work

Based on the literature analysed, the PhD student designed a research programme consisting of two independent stages in which she analyses the effect of the presence of natural jute and sisal fibres, followed by the ashes produced from the combustion of plant waste, on the properties of cement mortars, cement mortars with an aerating agent admixture and cement-lime mortars.

The author has clearly described the scheme of proceedings during the conducted research, from the making of composites to the determination of technological, mechanical and microstructural features of the matured materials. She has logically designed variants of subsequent composites so that it is possible to verify the thesis outlined in the dissertation. It should be emphasised that the Author made a significant contribution to the research, producing 27 variants of mortars with different compositions in terms of the proportion of mineral additives RHA, SCBA and lime powder and 14 variants of mortars containing a variable proportion of natural jute and sisal fibres as well as artificial ones based on polypropylene.

The presentation of the research results is clear and arranged in a logical sequence. All the results obtained are described and discussed. In practically all cases in the chapters dealing with the analysis of research results, the Author refers to the literature, sometimes confirming or contradicting the observed trends, which demonstrates her proficiency in the subject matter undertaken in the dissertation. She also tries to find

mechanisms explaining the described phenomena, which demonstrates her understanding of the relationships between the components of the tested materials.

A significant shortcoming is the lack of information regarding the actual homogeneity of the results obtained, particularly concerning the mechanical properties of the mortars. Assuming a confidence interval of 95%, she only assumed that the actual value of the analysed characteristic was within ±5% of the mean value, which may not necessarily be true, especially in the case of a random 3 or 6 measurements. On the one hand, in Chapter 5, "Testing methods", the PhD student does mention the determination of the coefficient of variation of selected features, but there is a lack of consequence in the rest of the dissertation.

In Chapters 8.7 and 9.7, the Author verifies the relationship between compressive strength and flexural tensile strength by rightly stating that for correlation coefficient R values above 0.9, a strong relationship between the features can be assumed. However, later in the analysis of the results obtained, the discussion is de facto about the coefficient of determination R2, which is an obvious oversight. Although there is an obvious mathematical relationship between the values of these coefficients, they relate to the description of different statistical parameters. It is also unclear whether all individual strength results or only the mean values were taken for the analysis.

The representativeness of the samples for studying the pore size distribution in materials, especially those containing fibres also seems quite debatable. Due to their small volume of approximately 2cm3, it is difficult to have confidence that the proportion of fibres will be averaged. In addition, it is also worth noting that, in the graphs containing the results of MIP tests, it is incorrect to directly compare the pore size distribution of materials with different specific densities (skeleton density) presented in units of mL/g. As a general rule, the porosity of materials is expressed in %, so the pore volume is related to the volume of the material tested.

4.4.Evaluation of the solution of a scientific problem

Undoubtedly, a valuable aspect of the dissertation is the broadening of knowledge regarding the implementation of sustainability in construction. The Author showed that it is possible to use natural fibres as well as ashes of plant origin, which not only reduces CO2 emissions as a result of limiting the amount of clinker used in cement mortars but also, in the overall balance, may lead to its partial sequestration during the vegetation period. As the Author has shown, a properly designed composition of cement mortars with natural fibres or plant-derived ashes can lead to improved mechanical properties of the composites.

5. Detail remarks

The Author has shown great attention to the editorial and editing side of her work. The structure of the subsequent chapters is logical and clear. The PhD student correctly operates the nomenclature characteristic

of building materials technology. The diagrams presented are clear and aesthetically designed. The following critical remarks do not lower the overall assessment of the dissertation.

1) In the literature review, there are places where the reporting of test results is imprecise, e.g. it is not clear what material is involved or what additive was used or under what conditions the composite was cured. There is also an incorrect use of units, e.g. for ash particle size or the working range of the testing machine.
2) The knowledge contained in Chapter 2 was mainly based on standards, which is not a complaint, and I even consider it right to make some kind of summary of criteria, divisions or assessed properties. However, in the reviewer's opinion, the standards should be consistently referred to when describing the individual characteristics of mortars or their types. Furthermore, the chapter lacks reference to any literature for a few paragraphs.
3) In subsection 3.2.2.1, "Physical properties of natural fibres", very little information is given about the physical properties of fibres, but there is a broad description of the influence of natural fibres on the properties of cement mixtures containing them. Thus, the title of the subchapter should rather be verified.

   In the same chapter, the PhD student, referring to the literature [22], claims in the first part of the paragraph that the presence of natural fibres reduces the workability of the mixtures, while in the summary it is stated that it nevertheless improves it as a result of water absorption due to the presence of lignin in the fibre structure. What is the Author's opinion on this issue?
4) A similar situation to the title of chapter 3.2.2.1 is encountered in chapter 3.2.2.2, "Mechanical properties of natural fibers". The title indicates a description of the mechanical properties of fibres, but in fact it concerns cementitious composites with natural fibres.
5) An analogous problem to that described above in points 3 and 4 occurs in the chapters on physical and mechanical properties of polypropylene fibres, i.e. 3.3.1.1., 3.3.1.2., and ashes, i.e. 3.4.2.1. and 3.4.2.2.
6) In Table 2 on page 14, the elastic modulus values have been formatted as dates, e.g. 6-Apr.
7) The stress-strain Fig. 1 on page 15, which is important for the characterisation of the properties of natural fibres, has not been interpreted or commented on by the Author. What relevant information for the dissertation did the Author intend to extract from it?
8) On page 20 in chapter 3.4, the same paragraph concerning the CO2-neutrality of bio-ashes appeared twice. The literature cited is different i.e. [60] and [61] and [62] and [63].
9) A very interesting, illustrative diagram on the chemical composition of mineral additives to cement was presented by the PhD student in the figure marked Fig. 2. However, I believe that knowing the chemical composition of the ashes formed from the combustion of organic substances discussed in section 3.4.1,

it would be beneficial to present them against the background of the generally known additives in the aforementioned Gibbs triangle.

10) In Table 3 in section 3.4.2.2, the trends presented are not commented on in any way by the Author in the text. It is not clear what conclusions the Author intended to draw from it.
11) In chapter 5.3., the information on the determination of the modulus of elasticity appears to be unnecessary, as the author did not plan to carry out such tests.
12) Chapter 6, "Materials", describes the components that constitute the binder of the composites produced - Portland cement CEM I of strength class 42.5 and a lime binder. While the author defined their chemical composition, the description lacked the basic characteristics of the selected cement, as well as information on the type of lime binder, whether it was used in the form of slaked lime or quicklime. Furthermore, the chemical composition is given in only about 90%. It is not known what constitutes the remainder of the lime binder.
13) In Table 6 in section 6.3 "Waste additives", the chemical composition of SCBA ash exceeds 100%, while for RHA a composition of about 80% is given. Also of wonder is the value for the specific density of both additives, which is the same at 2.43 g/cm3. Would not the three times higher carbon content of SCBA alter the value of this characteristic? How was the specific density of these materials determined and how was the carbon content determined?
14) In Chapter 6, "Materials", subsections 6.3.1. to 6.3.4. on the description of the properties of sugarcane bagasse and rice husk ash and natural and artificial fibres used in the research, provide an extensive literature analysis of their use in construction materials. It seems, therefore, that this otherwise interesting information should have been included in the literature analysis section rather than in the research section. In my opinion, this lacked a more complete characterisation of the components used in the mortars analysed.
15) Table 8, which is key to the dissertation, lacks the units in which the Author gives the proportions of the ingredients. It is also not clear for what volume the mix was designed. From an analysis of the proportions given, it does not appear that the compositions refer to 1m3 of material, as has most often been assumed in the literature. The varying w/c values for CM and CA suggest, a varying amount of water in the composition, which may make a comparative analysis of the results obtained difficult. It seems that the table should emphasise, e.g. in the form of an additional column, the constant value of the w/b ratio, where the components RHA, SCBA, LS should be included in the binder composition.
    In addition, Table 8 indicates the use of CEM II cement, whereas the previously described cement characteristics refer to CEM I. There was also an error in the composition of the CA reference composite. The reported w/c+L value should be 0.69, not 0.44. A similar error also appears in Table 9.

16) Due to the significantly different densities of natural S and PP fibres (d_{PP}=0.91g/cm3, d_S=1.58g/cm3), using them in the same mass ratio results in their significant variation in dosage by volume. It is difficult to compare their effects on the properties of the composites obtained.
17) Some of the statements in the conclusions seem too obvious or were not confirmed by the research, such as the increase in air content in mortar mixes after the addition of APA (Air entraining Plasticizing Admixture) or the fact that APA influences the formation of crystalline structures in the composite matrix.

6. Summary and final conclusion

The subject matter of the evaluated dissertation is up-to-date and is of interest to many researchers, which was confirmed by the analysis of the cited literature. The PhD student has contributed a great amount of new knowledge concerning the technology of building materials using components of plant origin, which is part of the trend of sustainable development in the construction industry.

As shown above, the dissertation of Ms. Jyoti Rashmi Nayak, M.Sc. meets the requirements of the Act of 20 July 2018. Law on Higher Education and Science (Journal of Laws of 2018, Item 1668, Article 187) in terms of i) the Candidate's general knowledge in the scientific discipline of civil engineering, geodesy and transport, especially in the sub-discipline of construction materials engineering, ii) the ability to conduct scientific work independently, iii) the solution of the scientific problem posed. Taking into account all of the above, I propose that the submitted dissertation should be accepted for public defence.

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