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POSSIBILITIES OF USING SULPHUR AND PLANT ORIGIN WASTE FOR LIGHT-WEIGHT AND THERMAL INSULATION CONSTRUCTION COMPOSITES

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Abstract. The article notes that not all types of plant origin waste are appropriate for being used as fuel or left for decay. The environment is polluted by noxious gas that is generated in the process of heating or decaying. Sulphur waste is produced at chemical and oil industry plants as well as during the treatment of emissions resulting from fuel combustion. Thermal energy is produced in the process of combusting plant origin waste; however, the emitted fumes are pollutants. A much more effective way for the use of this waste is proposed, i.e. to produce and use a new type of thermal insulation construction composites that would allow long-term heat saving buildings. The performed tests covered the properties of components and the influence of plasticisers on the plasticity and compressive strength of hardened sulphur. The common behaviour of sulphur and plant origin waste – sawdust, boon, straw – in composite was also analysed. All the properties necessary for thermal insulation materials were identified – compressibility, compression and bending strength, thermal conductivity. They have the same value as other classical thermal insulation materials.

Keywords: solid waste; physical pollution; environmental sustainability; sulphur and bio-waste; thermal insulation composite; properties.

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Introduction

With the development of civilisation, the range of application of natural resources in human life and activities was expanding. Upon reaching the present level, it has become necessary to utilise vegetal waste. Large amounts of waste are produced in the construction industry and agricultural production sectors – wood sawdust, chips, boon and straw. Large quantities of vegetation found in water bodies, such as reeds and bulrushes, remain unused.

In recent years, a large share of all cellulose raw materials and related wastes has been used as a source of energy produced by combustion.

A more versatile and effective use of such wastes for the creation of thermal isolation products would enable the reduction of thermal energy inputs. Major energy inputs go to the construction and heating of premises, and therefore, as investigations have revealed (Nanazashvili 1990; Kazragis *et al*. 2004; Kazragis, Gailius 2006; Zavadskas *et al*. 2009), the aforementioned waste can be used for the production of construction composite products with enhanced thermal insulation properties. However, this requires an appropriate binding agent. For the purpose of combining cellulose materials, i.e. composite inserts, cement slurry as a composite matrix poorly interacts with cement or requires special additives.

Cement is a material of high energy intensity; its production results in large amount of CO_2 emissions and, in addition, it is a zero waste material. Besides, according to Mechta (2009), Portland cement contains about 85% clinker and the manufacturing process releases 0.9 tonne of CO_2 per tonne of clinker. These numbers show that cement manufacturing is one among such leaders producing large amounts of CO_2 as transportation, power generation, treatment of oil and gas and steel manufacturing. This is a problem to reducing the production of the main component of cement clinker. According to CEMBUREAU, it is necessary to reduce the portion of clinker up to 60% and to replace it by other materials with binder properties, such as waste, various ashes, slag, and silicium microdust or to replace all cement by another binder material. At the same time, concrete with new

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binder materials must have more durability than with cement. Such materials are sulphur or its waste. Generally, the use of sulphur, instead of cement, for the production of construction products was started nearly 50 years ago (Vroom 1992). The main reason for this replacement was quick setting, water-proof, chemical resistance and mechanical strength (Vroom 1992; Weber *et al.* 1992).

Nature is rich in highly sulphurous ore deposits reaching even the surface. However, only around 20–25% of pure sulphur is obtained from this ore and large amounts are left non-segregated from the impurities of various countries. Currently, plants of many countries, such as those of mineral fertilisers producing sulphuric acid, oil, gas, and other chemical industry plants, generate large amounts of sulphuric waste containing 2–60% of elementary sulphur (Michailov *et al.* 1989; Orlowsky 1988, 1990; Sulphur and Sulfur Containing Wastes 1990; Komnitsas *et al.* 2001; Gracia *et al.* 2004; Krishnan, Freeman 2008). The same amount of elementary sulphur can contain sulphur waste of proper chemical plants and energy powers as in Lithuania.

During solid and liquid fuel combustion, sulphur dioxide (SO_2) and trioxide (SO_3) emit into air together with small particles and various compounds that are harmful to the human organism form on the ground (Peavy *et al.* 1985).

The first in-depth research into this new material, sulphur concrete, was started in the USA and Canada. In Lithuania, research on the use of sulphur for fine concrete was launched in 1988 (Gauronskis *et al.* 1990; Marčiukaitis 1994).

As investigations carried out by a number of authors (Paturoev *et al.* 1985; Czarnecki, Gillot 1989a, b; Michailov *et al.* 1989; ACI Committee 548 1993; Mohamed, El Gamal 2010) show, the use of sulphur, as a binding agent for concrete production, ensures such main mechanical and physical properties of concrete which nearly do not differ from those of cement concrete, and some properties, such as resistance to corrosion and water-proof capacity, are even much better (Table 1). However, its wider application was impeded by higher energy inputs as a temperature of 140 ± 5 °C is required for concrete components within the entire process of concrete production. A large amount of sulphur has to be melted. The melting temperature is 130 ± 5 °C. Specially heated stirrers are required in the production of heavy sulphur concrete. If industrial sulphur is used, fine aggregate particles are necessary (Czarnecki, Gillot 1989b; Mohamed, El Gamal 2009, 2010).

However, little research has been done into sulphur waste by using the plant origin waste as a binding agent in the production of construction thermal insulation composites. The advantage of sulphur in all areas of its use, when compared to other binding agents of construction materials, is its quick setting in a normal environment (12–20 h).

Table 1. Comparison of the properties of sulfur concrete and cement concrete

Properties	Parameters of concrete	
	Sulphur concrete	Cement concrete
Mass (kg/m^3)	2300–2400	The same
Short-term strength (N/mm^2)		
Compression	45–70	The same
Bending	10–12	Lower
Tension	5–7	Considerably lower
Compressive modulus of elasticity (N/mm^2)	$(3.5–5.0) \times 10^4$	Nearly the same
Poisson's ratio	0.20–0.25	Nearly the same
Frost resistance	Cycles	Considerably lower
Resistance to aggressive acids		Considerably lower

In order to improve the inefficient use of this waste and minimise the nature pollution, it is appropriate to develop the ways of using it in production and construction of construction products with reduced thermal conductivity by reducing energy inputs (Kazragis, Gailius 2006; Gravitis *et al.* 2010). Therefore, it is advisable to analyse the combining of these types of waste with different properties into a common effective construction composite, thus identifying one of the ways of a more effective use of nature- and air-polluting waste.

1. Components and methods of research into their properties

To produce binding agents containing sulphur, it is possible to use pure sulphur by adding fine particles of another type, also sulphur waste.

Currently, considering the need to reduce pollutant emissions and preserve nature, thousands of tonnes of mixes of various particles containing sulphur are caught in equipment cleaning gas emitted during fuel combustion. Sulphur waste is very diverse and, as data show, can be divided into three types:

(1) Sulphur with a large content of solid mineral impurities of different sizes which are similar to those of aggregates used for concrete ($12 \div 35$ mm).
(2) Sulphur with fine impurities, such as dusty sand ($0.12 \div 1.25$ mm).
(3) Sulphur with minor mineral particles, such as flour ($0.12 \div 0.32$ mm).

Sulphurous waste of a similar type is produced by some enterprises of our country, i.e. a crude-oil refinery in Mažeikiai, fertiliser plants in Kėdainiai and others which are related to the production and use of sulphuric acid. Large amounts of it can be collected in plants consuming much fuel and cleaning emitted fumes and steam. The properties of sulphur are not exactly identical; they depend on various factors (Lvov Ukraina 1990; Abdel-Jawad, Al-Qudah 1994; Mohamed, El Gamal 2010; Vlahovič 2011). A major impact is made by the concentration of H_2S. With its content increasing, the strength of sulphur is decreasing. Therefore, sulphur obtained from different sources or its waste should satisfy the following general criteria:

- Density – 2 100 kg/m^3;
- Strength (N/mm^2): compression 18–20; bending 5–7;
- Coefficient of temperature deformations 5.7×10^{-6} °C;
- Thermal conductivity (W/(m °C)) 0.27–0.28.

Our investigations used sulphur from Mažeikiai Crude Oil Refinery, i.e. grain sulphur, 99.01%; ash of different compositions, 0.4%; various acids, 0.01%; organic admixtures, 0.4%; humidity, 0.15% and other chemical elements, 0.03%. Sulphur was melted in a special electrically heated tank at a temperature of (140 ± 5) °C. In order to determine the physical and mechanical properties of hardened sulphur, specimens were produced and tested in accordance with the standard requirements.

The following plant origin wastes were used as inserts for a composite with sulphur matrix:

(1) Coniferous timber sawdust and chips. Length of the particles, 1–30 mm; natural humidity, 10–18%; bulk volume mass, 110–150 kg/m^3; and coefficient of thermal conductivity, 0.07–0.11 W/(m °C).
(2) Flax boon – dimensions of the particles up to $50 \times 3 \times 0.3$ mm; bulk volume mass, 110–120 kg/m^3; average natural humidity, 15–20%; water adsorption capacity, 220–240% (according to mass) and thermal conductivity of dried boon, 0.04–0.1 W/(m °C).
(3) Barley and wheat straw cut in 30–50 mm long pieces; humidity, 10–15%; bulk volume mass 35–50 kg/m^3 and water absorption capacity, 50–80%.

Plasticising agents of three known types (paraffin, thiokol and dicyclopentadiene) were used. The best plasticising agent (no. 1) out of these types and their mixtures was suggested. It also mainly consists of cheap production waste. Generally, the impact of various plasticising agents on the fragility (plasticity) of sulphur has been widely researched (Jordan *et al.* 1978; McBee *et al.* 1981; Darnell 1991). The impact of the accepted plasticising agent (no. 1) on the mechanical properties of sulphur was analysed in accordance with the adopted methodology.

The investigations of composite products were carried out using mixtures of different compositions with wood sawdust, flax boon and cut straw as inserts. Specimen formation technology was the following: dried inserts were placed in a special holey form and pressed up to the desired density by putting a mesh. The product was submerged into melted sulphur for 15–30 seconds, then left for 20–30 seconds for it to trickle and was placed in a setting place. Melted sulphur fully sets after 3–5 h. Plasticising agent no. 1 was used as the main plasticising agent 1, as preliminary investigations with other plasticising agents produced much worse results.

The main mechanical properties of nearly all construction materials are compression and bending strength, for thermal insulation materials also compressibility. Compression strength is closely related to tensile strength. Four cubes, $100 \times 100 \times 100$ mm, and four prisms, $40 \times 40 \times 200$ mm, were tested for the determination of each of the indicators. Prior to testing, their dimensions were specified and density of the specimen was identified, as presented in Table 2. Compressibility was determined by two methods: assuming that a load is equal to 2 kPa and that the

Table 2. Composition of mixtures and density of products

Composition no.	Product density (kg/m^3)	Component quantities (kg/m^3)				
		Sulphur	Plasticising agent no. 1	Boon	Straw	Sawdust
1	350	220	1.1	150	–	–
2	400	250	1.25	150	–	–
3	480	310	1.5	170	–	–
4	200	140	0.70	–	60	–
5	250	190	0.95	–	60	–
6	320	240	1.20	–	80	–
7	450	240	0.60	–	–	150
8	520	340	0.60	–	–	180

load accounts for 30% of failure load *F*. In accordance with this method, compressibility *S* is:

$$S = \frac{\Delta h}{h \cdot \sigma}, \quad (1)$$

where Δh – absolute compression at 0.3 failure load *F*; *h* – initial height of the specimen; σ – stresses when $\sigma = 0.3F/A$; *A* – specimen's area under compression.

The properties of any composite, particularly mechanical, are largely dependent on the cohesion between the matrix and inserts (Marčiukaitis 1998; Blankenhorn *et al.* 2001). The cohesion between sulphur, as a matrix, and plant origin waste, and inserts, was determined in two ways. Using wood inserts, the wood specimens, 100 × 100 × 30 mm, were produced and glued together with the sulphur binder at issue. Prior to gluing, they were dried to a constant weight. The second way – an indirect investigation of cohesion during the testing of composite elements by compression and bending in accordance with the nature of failure by examining the failure area with a microscope with a 150 times magnifying capacity.

For the purpose of determining the thermal conductivity of the products, the specimens, 200 × 250 mm, with a thickness of 66 and 80 mm (boons), 80 mm (straw) and 100 mm (sawdust) were made. They were made in accordance with the composition of components indicated in Table 2. Tests were carried out in accordance with the methodology that meets standard requirements.

2. Research results and their analysis

2.1. The properties of melted sulphur and its cohesion with inserts of plant origin

Sulphur was melted – its melt is highly penetrative and plastic in a liquid state. It, therefore, perfectly fills volumes composed of bulk or fibre thermal insulation materials binding them into a monolith.

During the setting, sulphur converts from amorphous into crystalline, therefore, its density and strength rapidly increase. Quick setting, either in contact with another material or in its own volume, results in the occurrence of internal stresses. As investigations carried out by other authors and by us show, sulphur is a fragile substance (Mohamed, El Gamal 2009, 2010). However, this property is enhanced either by plasticising a melted sulphur or adding the stabilisers of its structure (McBee *et al.* 1981; Darnell 1991; Gracia *et al.* 2004). However, the use of plasticising agents reduces the strength of sulphur. As the charts presented in Figure 1 show, the strength of plasticised sulphur depends on the content of the plasticising agent. With the amount of plasticising agent increasing, the strength is decreasing.

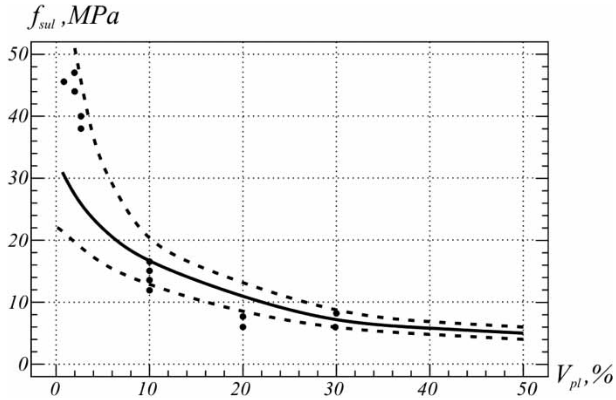

Fig. 1. Influence of plasticiser no. 1 on the compressive strength of sulphur

As the strength of thermal insulation materials is relatively low and the volume ratio of a binding component is small, a lower strength of a matrix does not have a significant impact. On the other hand, as investigations have shown, a plasticising agent proportion of 0.065–0.60% is sufficient to enhance the plasticity of sulphur. This also depends on the type of a plasticising agent. The quantity of plasticiser used for the production of our specimens is presented in Table 2. However, a plasticiser has a significant impact on the plasticity of hardened sulphur. This is proved by the investigations carried out by us (Fig. 2) and other authors (Jordan *et al.* 1978; McBee *et al.* 1981).

An important factor of the properties of composite materials is cohesion between the matrix (sulphur) and inserts (plant origin waste).

The theory of construction materials, including composites, covers not only a widely applied law of mixture but also a dependence approximating the dependence of their strength on a number of factors according to Nanazashvili (1990):

$$f_c = \frac{f_m}{\left(t_m / t_0 \right)^n}, \quad (2)$$

where f_c – composite strength; f_m – binder strength; t_m – average thickness of binder's film between inserts;

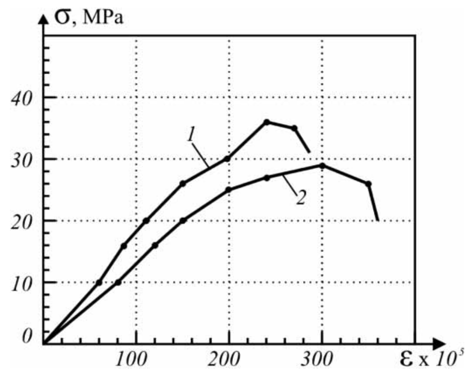

Fig. 2. Deformation character of hardened sulphur during compression: (1) non-plasticised, (2) plasticised

t_0 – thickness of a binder film of inserts of the optimum composition; n – indicator dependent on the physical and mechanical properties of inserts, their form, cohesion strength, film thickness, etc.

As the physical and mechanical properties of the aforementioned inserts do not differ much and have a similar geometrical form, only an impact of a film (layer thickness) on cohesion between inserts was investigated.

As the analysis of the curves in Figures 2 and 3 shows, when a thickness of the binder film is smaller, cohesion strength is bigger. This can be explained by the inter-molecular interaction of components in the pre-contact zone. The thinner a sulphur layer is, the relatively bigger its depth covered by this interaction. In addition, when a sulphur layer is thicker, greater internal stresses, and even micro cracks, occur when sulphur shrinks during the setting. This weakens relationships between both the components.

In all the specimens, failure occurred within the limits of the hardened layer of sulphur without affecting wood whose surface was polished. It should, therefore, be considered that such a surface corresponds to the smoothness of all straw and boon.

Cohesion among the components was analysed in an indirect way in accordance with the nature of surface that has disintegrated during testing.

As the analysis of specimen disintegration shows, cohesion between components depends on the surface of inserts: the surface of wood and boon is rougher than that of straw and, therefore, nearly no destruction takes place via the surface of component contact, while the surface of straw is much smoother and, therefore, disintegration most frequently takes place via the surface of contact.

2.2. Physical and mechanical properties of composite

The compressibility of light-weight and thermal insulation materials is closely related to strength as well as density.

As the obtained data about compressibility show (Table 3), the compressibility of the articles whose compositions are presented in Table 1 depends on the properties and quantities of components. The compressibility of composites with straw inserts exceeds 1%. However, it is nearly by three times below the compressibility of some mineral wool articles. The compressibility S of products with inserts of other types of plant origin is yet lower. For comparison, Table 3 shows the compressibility when calculating the value S (Eqn 1). As the comparison of compressibility results obtained by both methods shows, in both cases, articles with straw inserts have higher compressibility. This can be explained by a higher void content of straw structure and at the same time its lower density (kg/m^3).

Table 3. Compressibility of composites with components of sulphur and plant origin waste

Composition no.	Insert type	Compressibility	
		Percent	S (MPa^{-1})
1	Boon	0.05	0.25
2		0.04	0.18
3		0.02	0.11
4	Straw	2.05	12.05
5		1.80	9.00
6		1.60	8.00
7	Sawdust	0.02	0.095
8		0.17	0.083

This testifies to the fact that it is possible to obtain a thermal insulation building material which can resist higher pressure loads than a number of other thermal insulation materials used without infringing structural and thermal insulation requirements.

When testing compressibility up to 10% of deformation and continuing to apply a load, there was an identified compressive strength before destruction and, on the basis of bend specimen results, the bending strength and character of destruction were determined. The obtained results of strength identification are shown in Table 4.

As the data presented in Table 4 show, strength depends on the material density of products, as in the case of many other materials of low density.

As the analysis of the disintegrating nature of bending elements and surface shows, the failure nature of composite products with the matrix of sulphuric waste and inserts of plant origin waste is typical of the composites of other components with dispersive inserts (Marčiukaitis 1998). The performed investigations make it possible to point out three different features of failure which are also characteristic of other composites with dispersive inserts: failure via (1) matrix (sulphur); (2) a surface of contact between

Table 4. Compressive and flexural strength of composites made of sulphur and plant origin waste

Composition no.	Compressive strength (MPa)		Flexural strength (MPa)
	Deformation (10%)	Failure strength	
1	0.234	–	0.203
2	0.308	–	0.231
3	0.394	–	0.317
4	0.019	0.025	0.023
5	0.023	0.027	0.048
6	0.041	0.050	0.075
7	–	0.424	0.295
8	–	0.504	0.340

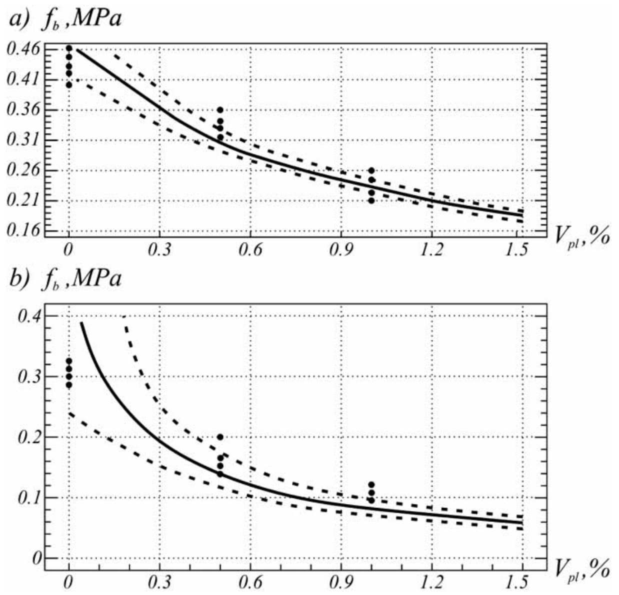

Fig. 3. Influence of plasticiser's quantity on the strength of binder's film during splitting, when film thickness is: (a) 0.5 mm, (b) 5 mm

the matrix and inserts and (c) inserts and matrix (Fig. 4).

As the thermal conductivity of sulphur λ (0.27–0.28 W/(m °C)) is lower than that of cement slurry, it was necessary to carry out an analysis of the thermal properties of composite with sulphur matrix and interests of plant origin waste. This is also predetermined by the thermal conductivity λ_c of obtained composite material. There is a direct relationship between this characteristic and heat resistance R (m°C/W). During experimental investigation, the thermal conductivity λ was obtained according to the experimental results of heat resistances of specimen R_{sp} and of contact between specimen and source of heat R_k. Then $R = R_{sp} - R_k$, as it is known $R_{sp} = \Delta T / q_{vid}$ and $\lambda = \delta / R_{sp}$. According to this data and other known suggestions (Fokin 2006), formula for the calculation of thermal conductivity of investigated composite material is:

$$\lambda_c = \frac{\delta}{\Delta T \big/ q_{vid} - nR_k}, \qquad (3)$$

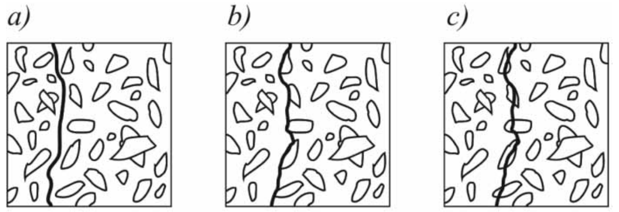

Fig. 4. Types of destruction of specimens made of sulphur and plant origin inserts: (a) typical of boon and sawdust inserts, (b) straw, (c) sawdust

where δ – specimen thickness (m); $\Delta\ T$ – difference in surface temperatures (°C); q_{vid} – density of the average heat flow passing through the specimen (W/m^2); R_k – thermal resistance of contact between the specimen and the source of heat (m^2 °C/W); n – the number of contacts.

As proved by investigations, the thermal conductivity of a composite with chaotically arranged inserts can also be determined analytically with sufficient accuracy. One of the best of such analytical equations according to G. Dulnev is the following (Marčiukaitis 1998):

$$\lambda_c = \lambda_m \left[1 - \frac{3\ V_{inc}\left(1 - \frac{\lambda_{inc}}{\lambda_m}\right)}{2 + V_{inc} - \lambda_{inc}\left(1 - \frac{\lambda_{inc}}{\lambda_m}\right)} \right], \qquad (4)$$

where λ_c – thermal conductivity of composite; λ_m – thermal conductivity of matrix (sulphur); λ_{inc} – thermal conductivity of inserts (boon etc.); V_{inc} – relative volume of inserts.

As the comparison of experimental and calculated values of thermal conductivities λ_c obtained using Eqn (4) (Table 5) shows, the analysed composites made of waste have better properties than solid mineral wool boards whose production requires big thermal energy inputs. As the comparison of data obtained by other authors (Nanazashvili 1990; Kazragis, Gailius 2006) when using cement slurry as a binding agent shows, the thermal conductivity of the composite in question is nearly twofold lower. In addition, the proposed material requires less deficit binding agent and utilises production waste which pollutes the environment (Fig. 5).

Naturally, the authors' investigations did not cover various technological factors which have an influence on the physical and mechanical properties of the composite material of production waste in question. However, as proved by these investigations, production waste can be used in the areas causing lesser environmental pollution and producing a higher energy effect.

Table 5. Thermal conductivity of the composite material made of waste

Waste (insert) type	Density (kg/m^3)	Conductivity with sulphur matrix	
		λ_{teor} (W/(m °C))	λ_{eks} (W/(m °C))
Boon	350	0.051	0.043
	400	0.055	0.040
Straw	200	0.042	0.035
Sawdust	450	0.075	0.076

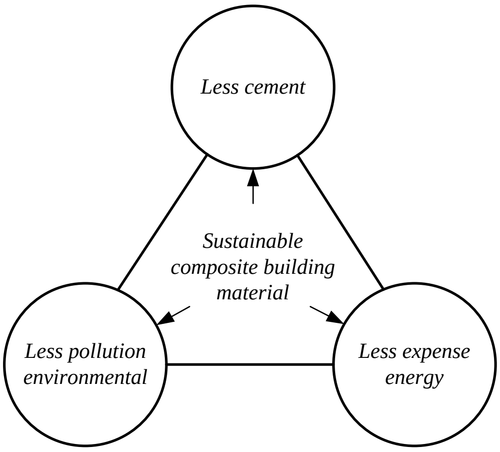

Fig. 5. Model of sustainable composite material using sulphur and plant origin waste

Conclusions

As the investigations carried out with sulphur and plant origin waste polluting the environment show, the production of composite construction of light-weight and thermal insulation materials and the products of this waste are more effective alternatives to combustion (fuel). Proper selection of composites' compositions makes it possible to obtain zero waste, energy-saving products, which fully utilise sulphur and plant origin waste and does not pollute the environment, which happens when it is combusted or left to decay.

The composite material obtained from the analysed waste polluting the environment is, by a number of its properties, equal to the currently widely used thermal insulation materials whose production requires larger energy inputs. Such important parameters of thermal insulation materials as compressibility and thermal conductivity have the same value as other classical thermal insulation materials used for building structures.

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