Opposition Review

of Doctoral Dissertation Thesis

Name: Kinetic Study of Selected Combustion Process

Author: Ahmed Maraden, M.Sc.

1. Thesis goal definition

The submitted dissertation thesis, consisting of 137 pages, has been formally elaborated in a common layout. The table of content contains a list of symbols, tables and figures and a list of the literature used, 172 items in total. The list of literature does not only include papers concerning new energy materials, proposed and tested according to the mentioned literature as a part of solving modern high-energy propellants based on two-component solid fuels (nitrocellulose + nitro-glycerine), or with new types of energy nitrocellulose gelling agents (BuNENA and DNDA57), but it also includes older papers concerning a study of burning of two-component TPH with an addition of nitramines and basic papers in the field of theory of combustion.

The thesis stipulates a series of goals both in the field of laboratory preparation of the solid fuels, and in the field of detection of characteristics of the prepared compositions, especially the dynamic decomposition, sensitivity, chemical stability and combustion rate. Three determination methods were used for the determination of dependence of the combustion rate on pressure, and results were compared. Considering the scope of the problems studied, the author selected two nitramines (RDX and BCHMX) and a mixed (heterogeneous) TPH based on a polybutadiene binder (HTPB) with ammonium perchlorate (AP) as an oxidizing agent. A part of laboratory works was implemented in laboratories and testing facilities of Explosia and OZM.

The theoretical part specifies a basic overview of the theory of solid masses, raw materials used and their characteristics. The main focus in on the technology of preparation of cast TPH both based on a two-component powder mass, including the preparation of components and mixed TPH with a rubber-type binder. The following part specifies basic terminology in the theory and catalysis of TPH combustion, and describes methods for measuring the rate of combustion and the determination of other characteristics (DTA, DSC). This part includes an overview and basic theory of all methods used for the preparation of TPH samples and their testing.

It is clear, as said above, that the planned scope of work is large and the experimental part, especially the method development and the preparation of samples, is also highly time consuming.

2. Selected manner of solution, its originality and implementation

It is obvious from the specified overview that the planned experimental works rely on laboratory preparation of samples of solid fuels, subsequent determination of selected characteristics and their evaluation. The procedure of experimental works is described in Chapter 2. The selection of raw materials and their characteristics (page 50) specifies their origin and basic properties of the used raw materials. For commercially available raw materials, their basic characteristics and potential own manner treatment are specified (AP — sieving).

However, for some raw materials only their origin is specified (Explosia), but a lot of important characteristics related to the studied problems of an effect of additives on the characteristics of the prepared modified TPH are missing.

As an example, it is possible to give a size and distribution of particles in RDX and BCHMX. This parameter affects the rate of combustion and sensitivity of the prepared TPH samples. A composition is only specified for the used comparative samples of commercial TPH. Any data on the determined or calculated characteristics – explosion heat, density, or specific impulse etc. are missing. It is not mentioned whether it is a composition according to dosing or the values determined analytically (e.g. data on weight and specification of the burning catalyst are missing at samples 1331 and 1333). These samples are used as reference ones for validation of individual, further in the thesis discussed methods of combustion rate determination and these data are important for precise characterisation of the used TPH as they affect the combustion rate.

The experimental part describes individual stages of preparing the raw materials for the cast TPH — granules produced using the preparation technologies of NC powders and a brief description including photographs of laboratory equipment. This part is well and lucidly elaborated.

The next part specifies detailed laboratory procedures for preparing the cast TPH, including the composition and preparation of granules and the procedure of mixing, casting and curing of the TPH mixture. With regard to the fact that this type of TPH has not been prepared in the Czech Republic before, this part is also significant for potential further continuation of works.[1]

In the tables, page 58 and 60, there are compositions of granules as well as compositions of resulting TPH. It can be noted to the table on page 60 that it would be suitable to add at least basic energy characteristics of the prepared TPH, i.e. a calculation of basic thermodynamic characteristics and/or provide a table of composition according to the chemical analysis and the determined parameter values of the prepared samples – explosion heat, density, stability at l00°C etc. Considering that a meaning of preparing and developing modified two-component TPH (or mixed TPH) is to increase an energy content and thus power, energy characteristics are one of important parameters to assess the prospects of prepared compositions.

It is similar with the described samples of mixed TPH based on HTPB, where the table on page 62 does not provide even basic characteristics of the prepared and further monitored samples (explosion heat, density). A detailed specification of the used active coal is also missing (Table 2.5, sample C 1) – is it carbon black or actually active coal? The use of carbon black or active coal is unusual for the known compositions of mixed TPH; the mechanism of burning of these TPH differs from the burning of a homogeneous base both of classical, and modified two-component TPH, and the effect of potential carbon, in general, is therefore minimum for this type of TPH, as confirmed by the experiments. In this regard, it would therefore be more methodologically suitable to compare an effect of catalysts on pure TPH without any addition of combustion modifier.

1 In the past, cast and absorbed TPH were developed on the basis of spherical powders in the Research Institute of Industrial Chemistry (VÚPCH). They were not applied in practice and development works were stopped.

3. Solution and results achieved

The thesis provides new knowledge in the field of laboratory preparation of samples of modified two-component TPH, casting technologies. The developed procedure enables preparing samples for the study of various additives with regard to new energy materials and/or other additives such as aluminium, combustion catalyst etc. The proposed and verified procedure of preparation enables a reproducible preparation of samples, which can be used both for studying properties of new TPH with regard to the interaction of energy additives and a basic binder, and basic pyrostatic and ballistic tests for the verification of possibilities for the use of the proposed compositions for practical solving of propellants for rocket engines.

With regard to the verification of the prepared samples, the thesis focuses on two main areas – a study of the thermal decompositions of the prepared samples and a comparison of the effect of concentration of individual additives, and on the determination of dependence of the combustion rate on pressure, again for various concentrations and combinations of additives. The main contribution of the thesis is in this field.

The TPH combustion rate measuring method and subsequent evaluation of results is based on the classical method of using a test rocket engine and the use of a newly introduced procedure for the determination of combustion rate of rocket TPH in a ballistic bomb (Stojan's bomb) and a new modification of this bomb which corresponds to the Crawford's bomb. These two methods also significantly reduce the necessary number of determinations and reduce the time of the whole process.

A contribution of the assessed thesis is the comparison of these methods of determination of the combustion rate dependence on pressure performed for several samples of the classical homogeneous TPH which proved that the obtained results are in fact identical and each of the methods used thus provides credible results.

Furthermore, a combination of energy plasticizers (BuNENA and DNDA 57) was verified and especially of BCHMX nitramine whose use has been proposed and studied according to the mentioned literature especially as a high-energy additive pro special plastic blasting explosives. The usability of this substance has been experimentally proven as a part of modified TPH. In terms of effect, BCHMX is comparable with RDX but an increase of the energy content of the modified TPH can be expected. This is also an important contribution of the thesis.

Results published in individual parts of the thesis are presented especially in the form of tables and charts. I consider the presented discussion over results in individual parts to be a real evaluation based on author's own results and it is not in contradiction with data published in literature.

4. Formal level of the thesis

As already mentioned, the thesis has been formally elaborated in a highly precise manner, it contains all the necessary elements, such as a list of tables and figures, and especially a relatively extensive overview of literature (with specified references in the text). The graphic processing is lucid, inserted figures of apparatuses and instruments used, especially in the part devoted to the sample preparation, are suitable and clearly demonstrate the text.

5. Questions and comments

I have already mentioned the main comments in the previous parts of the review. In the conclusion, the author proposes topics for further continuation of works. The first point mentioned is an effect of organic additives (nitramines) on mechanical properties of TPH. I consider this to be one of the basic criteria for prospective spreading of the use of this type of TPH.

Another proposed direction of research is thermal sensitivity of modified TPH with BCHMX and/or further increase of power by adding nano Al, which should result in further increase of explosion heat, and therefore there is a presumption of an increased power of TPH modified in this way

However, the research to the study of an effect of particle size of the proposed CMCB combustion additives on TPH and ballistic properties of this group of propellants should be considered decisive.
Then, it is a question of quality and a method of quality inspection of the cast TPH filling charges, which relates to the technology of sample preparation.

My question concerns non-destructive testing of the produced samples, i.e. how homogeneity of the prepared samples was inspected.

6. Overall assessment and recommendation

I assess the thesis to be valuable in general. The author prepared numerous samples of modified two-component TPH and for each of the samples, he determined basic characteristics, of which especially the verification of temperature characteristics, basic sensitivity and the assessment of an effect of additives on combustion rate are of the highest importance for potential continuation of works. The results of the thesis are in line with the previous knowledge published in literature and suitably extend the knowledge of the effect of crystalline nitramines on parameters of modified TPH.

I recommend the thesis to be defended.

Pardubice, 23 October 2018

Doc. Ing. Ladislav Lehký, CSc