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Research paper

Investigation of the Parameters on the Preparation and Characteristics of a High-Energy Spherical Propellant

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Abstract: Spherical propellants are widely used in handgun ammunition due to their favorable ballistic and manufacturing properties. Enhancing their energy content is a key objective for improving bullet velocity and overall performance. In this study, a high-energy spherical propellant was developed via an emulsion-based method, optimizing the NM/EAc (nitromass/ethyl acetate) ratio and solvent removal conditions. The optimal formulation was identified at an NM/EAc ratio of 1/2.5 with 50% solvent removal, resulting in spherical particles primarily in the 0.125-0.315 mm range (80 wt.%). The final product exhibited a combustion heat of approximately 998 cal/g, representing a 9.07% increase over conventional spherical propellants. Ballistic testing with 7.62×25 mm Tokarev cartridges showed that coating the propellant with 2% ethylcentralite improved bullet velocity by 8.9%. Importantly, the enhanced energy content did not compromise key physical characteristics such as specific gravity, bulk density, or chemical stability. These results demonstrate that careful adjustment of formulation parameters can lead to higher-performance spherical propellants suitable for small-caliber applications.

Keywords: nitromass, energy, ratio, ethyl acetate

1 Introduction

Spherical propellant is a type of granular propellant with a spherical, near-spherical, or flattened form, based on nitrocellulose (NC), nitroglycerin (NG), and different additives. The spherical propellant can be classified into double-base and multi-base types. A typical double-base spherical propellant contains two energetic components, approximately 80-90% NC and 10-20% NG, with the other components comprising 1-3% [1-3]. Additionally, explosives or metal powders can be incorporated into the double-base spherical propellant to create triple-base spherical propellant with characteristics tailored to specific applications [4]. Among these types, the double-base spherical propellants are the most widely used. Several commercial propellants have been made available in the market by Explosia such as D020-01, D036-08, D063-03, and D073-02, which are used in various ammunition types, including 40×53 mm grenades, 9×19 mm NATO rounds, 7.62×39 mm, and 5.56×45 mm cartridges [5]. To improve bullet velocity, one approach is to enhance the characteristics of the propellant, including its energy content, morphology, and combustion behavior. In this study, a double-base spherical propellant is developed from NC, NG, and additives (centralite, graphite, lead phthalate, *etc*.) by increasing the ratio of NG to NC.

The industrial production of spherical propellant employs various methods, including emulsion techniques [6-9] and extrusion methods [10]. Of these, the emulsion method is the most commonly used. According to this emulsion method, the mixture of raw material components (nitro-mass, NM) of the spherical propellant is dissolved in a hydrophobic organic solvent (ethyl acetate, EAc) at a temperature gradually increasing from 40 to 65 °C to form a slurry. This slurry is then dispersed into an emulsion in water using mechanical stirring and stabilized with a surfactant at 65-68 °C. Next, the organic solvent is removed by distillation at progressively increasing temperatures (from 72 °C to 100 °C), combined with the addition of a dehydrating agent, resulting in nearly complete removal of EAc and the formation of spherical propellant particles. These propellant granules can then be coated with centralite at around 75-80 °C, graphitized, and flattened. Depending on the composition of the raw materials, factors such as solvent ratio, stirring speed, surfactant concentration, and dehydrating agent content can be adjusted to control particle size and density. In the extrusion method, single-base flattened spherical propellant is produced by extruding the material through a cylindrical die and cutting it into particles of a specific size, yielding flattened spherical particles with dimensions of 1.6×(2.3-2.5) mm. To develop a novel high-energy spherical propellant using the emulsion method with a solvent system comprising EAc and water, certain technological parameters

need to be optimized. To date, no published studies have provided viscosity data for the NM/EAc during the particle formation and solvent distillation stages.

For spherical propellants, key characteristics of interest include morphology, particle size, density, bulk density, combustion heat, and chemical stability. The typical particle size ranges from 0.2 to 3 mm in diameter, with a combustion thickness of approximately 0.25 mm [10, 11]. Combustion heat is a critical parameter used to assess the energy efficiency of the propellants, with values typically ranging from 900 to 1070 cal/g for conventional spherical propellants. The double-base spherical propellants generally have a density exceeding 1.5 g/cm³ [2, 3, 12]. Enhancing the energy content of propellants enables higher projectile velocities when using the same charge weight within the cartridge chamber. Depending on the application, the characteristics of spherical propellants can be adjusted by modifying their composition, particle size, and density.

Currently, no studies have reported the successful development of a high-energy spherical propellant. This paper aims to optimize key manufacturing conditions for propellant and investigate and evaluate its key characteristics.

2 Experimental

2.1 Materials

Nitromass BW (NM-BW) contains 80% NC (with 13.26% of nitrogen content) and 18% NG. Nitromass NB (NM-NB) is a mixture containing 59% NC (with 12.20% of nitrogen content), 39.5% NG, and 1.5% methylcentralite. The NM-BW and NM-NB, graphite powder, and lead phthalate were supplied by Chemical Company 95 (Vietnam). Ethyl acetate (EAc), sodium sulfate, and sodium carbonate were purchased from Xilong (China). Arabic gum was sourced from France.

2.2 Methods

2.2.1 Determination of NM viscosity in EAc solvent

The viscosity of the NM/EAc mixture was determined using a Brookfield DV-E Viscometer equipped with spindle 64. NM was dissolved in EAc at the selected ratio in a sealed glass beaker, stirred, and thermally stabilized at 65 or 73 °C.

2.2.2 Preparation of spherical propellant

The spherical propellant was prepared by an emulsion method using a 1000 mL three-necked flask. The process consisted of the following main stages: dissolution, dispersion, solvent distillation, dehydration, stabilization, drying, and coating.

In the dissolution stage, water-containing NM (NM, 50%) was dissolved in the EAc at an NM/EAc mass ratio ranging from 1:3.5 to 1:5. The mixture was maintained at 40 °C for 2 h, followed by a gradual increase in temperature to 65 °C over 4 h, under continuous stirring at 500-600 rpm.

During the dispersion stage, preheated water (65 °C) was added to the flask at an NM/water mass ratio of 1:(3.0-4.5). Emulsification was carried out at 600 rpm. To stabilize the emulsion, a 20% aqueous solution of Arabic gum was added, and the temperature was maintained at 65-68 °C for 30 min.

The first solvent removal was performed by distilling approximately one-third of the initial EAc content at 72-74 °C. In the dehydration step, sodium sulfate (5% of the total water mass) was introduced at 71 °C, and the mixture was held for 2 h. Subsequently, the temperature was increased from 72 to 78 °C over 4 h and maintained at 78 °C until no further solvent evaporation was observed (second solvent distillation).

Neutralization of residual acidity was carried out using 100 mL of a 2% sodium carbonate (Na_2CO_3) solution. The resulting emulsion was washed twice with hot water and three times with cold water to remove any remaining impurities.

The semi-finished propellant was dried in a convection oven at 60 °C for 6 h. Surface coating was then applied by treating the dried granules with 1-4% centralite. Finally, 0.3% graphite was added as an antistatic agent, and the granules were rolled to a final burning layer thickness of 0.10-0.12 mm using a rolling mill.

2.2.3 Determination of propellant particle size

The particle size distribution of the propellant was determined using sieves of 0.125, 0.200, 0.315, and 0.425 mm. The mass of each particle size fraction was weighed, and the percentage was calculated using the formula:

$$Zp_i(\%) = \frac{m_i}{\sum m_i} \times 100 \quad (1)$$

where Z_{Pi} is the percentage of each particle size fraction (in %) and m_i is the mass of fraction i.

2.2.4 Determination of specific gravity

The specific gravity of the sample was determined using a 50 mL pycnometer with distilled water as the medium at ambient temperature and corrected to 20 °C. The specific gravity (in g/cm^3) of soil is determined using Equation 2.

$$\rho = \frac{(m_2 - m_1).\rho^t}{(m_2 - m_1) - (m_3 - m_4)} \quad (2)$$

where m_1 is the mass of empty pycnometer, m_2 is the mass of the pycnometer with dry sample, m_3 – is the mass of the pycnometer and sample and water, m_4 is the mass of pycnometer filled with water only and ρ is the specific gravity of sample.

2.2.5 Determination of bulk density

The bulk density of the sample was determined following Method 201.1 of MIL-STD-650 [13] using a standard cup with a volume of 30 mL.

2.2.6 Determination of combustion heat

The combustion heat of the propellant was measured using a Parr 6200 calorimeter, following Method 404.1 of MIL-STD-286 [14].

2.2.7 Determination of chemical stability

The chemical stability of the propellant was assessed using a Vacuum Stability Tester, following the STANAG 4556 standard at 100 °C for 40 h [15, 16].

3 Results and Discussion

3.1 Investigation of the viscosity of the NM/EAc on particle formation and solvent distillation

In the manufacturing of spherical propellants using an emulsion system, product yield, particle shape, and size depend on the component ratio in the system (NM/EAc/water), stirring speed, surfactant concentration, and dehydrating salt concentration. In this experiment, a new NM with different characteristics was selected to improve the energy output of the propellant. When altering the raw material NM, the viscosity and rheological properties of the propellant slurry will change at various stages, affecting product shape, size, and density if technological parameters remain unchanged. Thus, the viscosity of the propellant slurry (NM/EAc ratio) is a crucial technological factor during particle formation, solvent distillation, and dehydration when changing raw materials.

To maintain the existing technological conditions, the viscosity of the propellant slurry with the new NM should match that of the traditional formulation at the respective processing stages. Since particle formation occurs at 65 °C, the viscosity of the propellant slurry at this temperature needs to be examined.

In traditional spherical propellant manufacturing using NM-BW, the NM-BW/EAc ratio is typically between 1/3.5 and 1/5. In this experiment, the viscosity of NM-BW/EAc and NM-NB/EAc was determined according to section 2.2.1. The viscosity of NM-BW/EAc was measured at ratios of 1/3.5 to 1/5, while NM-NB/EAc was measured at ratios from 1/3.5 to 1/1.25. The results at 65 °C are shown in Figure 1.

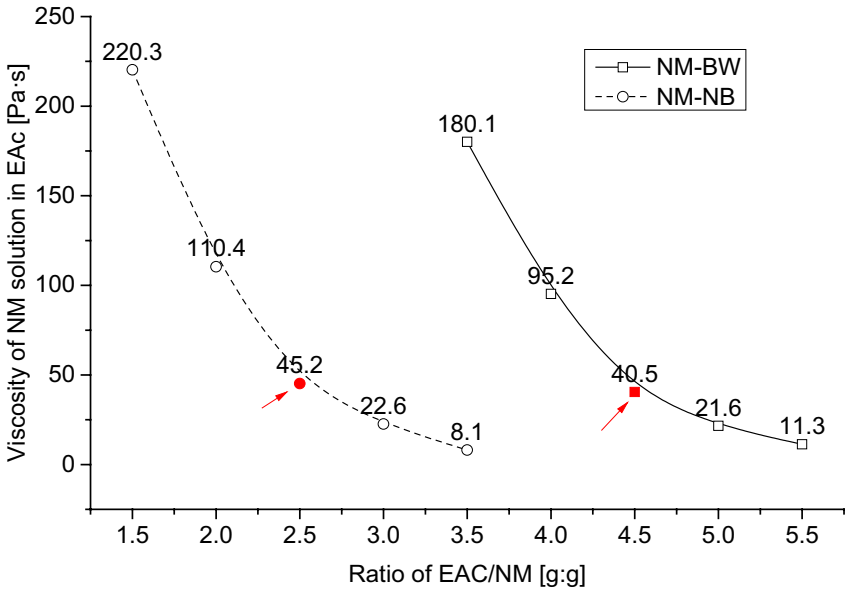


Figure 1. Effect of EAc/NM ratio on viscosity of NM solution in EAc at 65 °C

For conventional spherical propellants, NM-BW viscosity during dispersion ranges from 11.3 to 180.1 Pa·s (Figure 1). In practice, NM-BW/EAc is typically set at 1/4.5, corresponding to a viscosity of approximately 40.5 Pa·s. Based on Figure 1, the NM-BW viscosity at this ratio is 45.1 Pa·s, leading to the selection of an NM-NB/EAc ratio of approximately 1/2.5.

After particle formation, solvent distillation is conducted at a temperature range of 72-74 °C. The viscosity of the NM/EAc phase initially decreases with rising temperature but subsequently increases as the solvent evaporates. Understanding this viscosity change is essential for determining the required solvent removal. The viscosity of NM-BW and NM-NB in EAc was examined at 73 °C across NM/EAc ratios of 1/(3.5-5) and 1/(1.25-3.5), respectively. The results are shown in Figure 2.

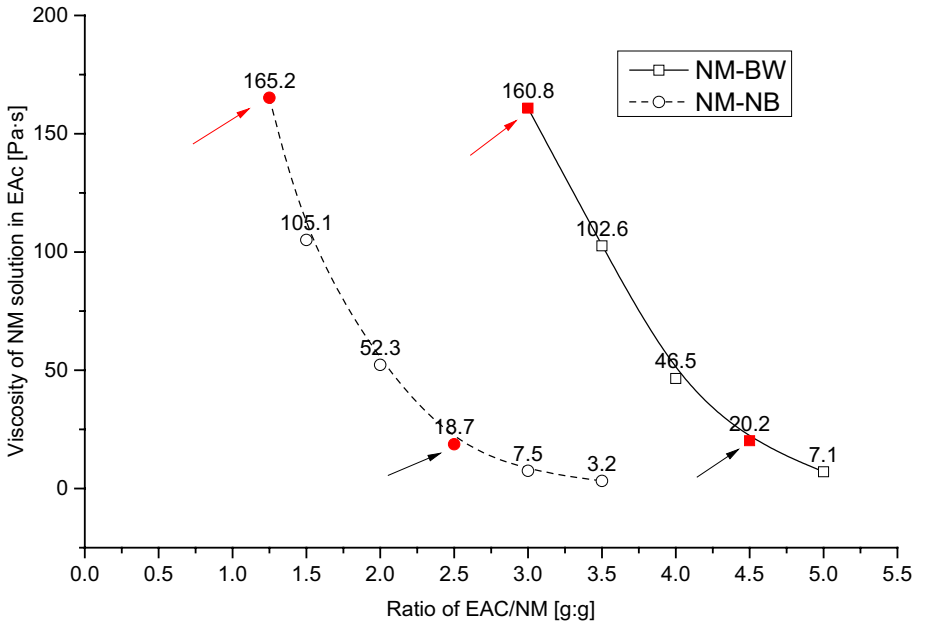


Figure 2. Effect of EAc/NM ratio on viscosity of NM solution in EAc at 73 °C

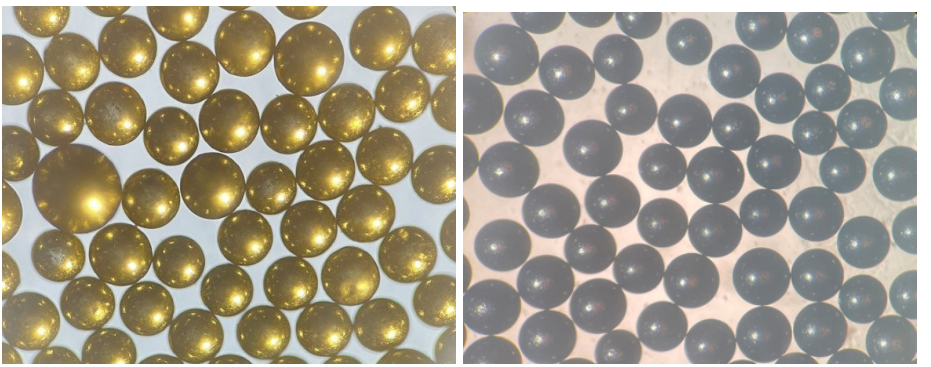
Results indicate that at solvent distillation temperature (73 ±1 °C), NM-BW/EAc viscosity decreases to 20.2 Pa·s, while NM-NB/EAc reaches 18.7 Pa·s. The greater viscosity reduction in NM-NB/EAc is acceptable and does not require stirring speed adjustments. When NM-BW/EAc reaches 1:3, viscosity rises to 160.8 Pa·s, corresponding to 33% solvent removal by the end of distillation. Thus, NM-NB/EAc must reach a 1:1.25 ratio to achieve equivalent viscosity, requiring 50% solvent removal.

To manufacture high-energy spherical propellant from NM-NB, the NM-NB/EAc ratio should be 1/2.5 during particle formation, with 50% solvent removal.

3.2 Technical characteristics of high-energy spherical propellant

To confirm these findings, a propellant sample was produced under the selected conditions, and its properties were examined. The raw material composition and processing conditions influence product characteristics, particularly shape, size, density, bulk density, and combustion heat. After production, these characteristics were assessed.

Surface morphology affects bulk density. High-energy spherical propellant samples were observed under an optical microscope. After graphitization, the propellant exhibited the morphology shown in Figure 3.



(a) (b)

Figure 3. High-energy spherical propellant from NM-NB before (a) and after (b) graphitization

The resulting spherical propellant exhibited a uniform dark yellow color with a smooth surface and spherical shape, indicating that initial technological adjustments were appropriate.

Particle size determines propellant applicability in various weapon systems. Factors such as NM/EAc viscosity, component ratios, stirring speed, and surfactant concentration affect particle size and distribution. In this experiment, the dispersion stirring speed was fixed at 600 rpm while other parameters remained unchanged. Particle size distribution results are shown in Figure 4.

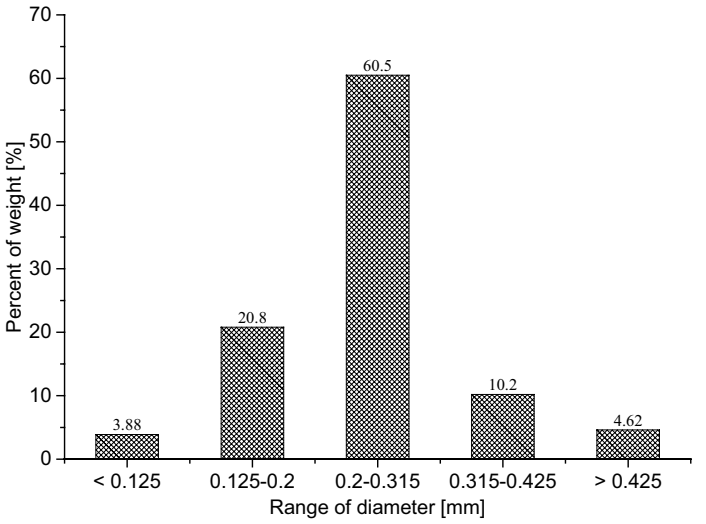


Figure 4. Particle size distribution of high-energy spherical propellant from NM-NB

Results indicate that over 90% of high-energy spherical propellant particles range from 0.125 to 0.425 mm, with 60.5% in the 0.2-0.315 mm range. The combined fraction of 0.125-0.315 mm accounts for 80% of the total mass.

Propellant quality is further assessed via specific gravity, bulk density, combustion heat, and chemical stability. Results for graphitized samples within the 0.125-0.315 mm range are shown in Table 1.

Table 1. Technical parameters of high-energy spherical propellant

Particle size [mm]	Specific gravity [g/cm³]	Bulk density [g/cm³]	Combustion heat [cal/g]	Chemical stability by VST [cm³/g]
0.125 to 0.315	1.58	0.95	1126	0.76

Within this size range, the specific gravity, bulk density, and chemical stability are comparable to previously published spherical propellants. In practical military applications, long-term storage stability of the new propellant formulation is very important. As shown result, a VST value of 0.76 cm^3/g indicates a low rate of gas evolution (mainly NO_x) at 100 °C over 40 h, suggesting a slow rate of decomposition and acceptable short-to medium-term chemical stability. According to STANAG 4582 [17] and explosives literature such as Meyer *et al.* [18], double-base propellants are considered chemically stable if the VST value is below 2.0 cm^3/g – the threshold beyond which decomposition becomes hazardous and disposal is recommended. In practice, double-base formulations with VST values in the 0.5-1.0 cm^3/g range are still considered safe for use and storage under standard conditions (20-25 °C, low relative humidity), with shelf lives of up to 10-15 years. When compared to commercial spherical propellants such as NC/NG/DNT or NC/NG/DEP-based systems, which typically show VST values in the range of 0.65-1.10 cm^3/g [3], the tested propellant with 0.76 cm^3/g falls well within the safe range and exhibits comparable or superior chemical stability. Nevertheless, it is important to note that the VST only reflects thermal decomposition under ideal laboratory conditions. A comprehensive assessment of long-term stability should also include accelerated aging tests (*e.g.* at 65 °C), identification of decomposition products *via* HPLC or FTIR, and studies on compatibility with packaging materials (*e.g.* copper, steel, epoxy resins). Therefore, while the obtained VST value of 0.76 cm^3/g is a strong indicator of chemical stability, further investigations are necessary to confirm long-term storage performance, particularly for applications involving harsh environmental or military conditions.

Notably, combustion heat improved significantly, reaching 1126 cal/g, a 23.1% increase over conventional spherical propellant. Thus, the energy of the propellant has been significantly improved, leading to the ability to improve the velocity of the bullet when using this propellant. However, to match the specifications of the bullet type used, it is necessary to implement a solution that ensures consistent barrel pressure during firing.

To evaluate the effectiveness of the propellant after energy improvement, this propellant was loaded into 7.62×25 mm Tokarev cartridges, replacing the currently used propellant (P45) with an equivalent loading amount. The results showed that the bullet velocity increased significantly; however, this was accompanied by a rise in pressure (as shown in Table 2).

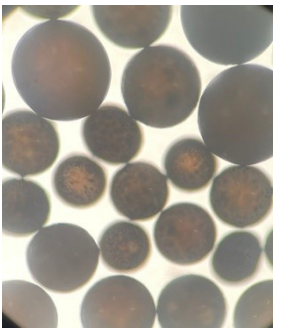


Figure 5. Microscope image of coated and flattened propellant based on NM-NB

To address the issue of maintaining barrel pressure during firing, this propellant was coated with 1% to 4% centralite No. 1 as a compound for progressive burning and flattened to create a burning thickness of 0.1-0.12 mm. The results are presented in Figure 5 and Table 2.

Table 2. Firing results of the propellant on the K54 pistol for loading amount of 0.56 g

Percent of centralite [%]	Combustion heat [cal/g]	Bullet velocity [m/s]	Pressure [atm]
0	1126	495	2317
1	1068	492	2115
2	**998**	**488**	**1865**
3	952	472	1658
4	925	451	1501
P45[(a)]	915	448	1769

(a) refers to the traditional porous propellant used in the charge of the 7.62×25mm Tokarev cartridge

Using the high-energy spherical propellant significantly improved the bullet's velocity. With the same loading amount, the bullet velocity using the high-energy spherical propellant reached 495 m/s, an increase of 47 m/s (10.5%). However, the barrel pressure also rose to 2317 atm, an increase of 548 atm (30.98%). When using propellant coated with 2% ethylcentralite No. 1, the barrel pressure reached 1865 atm, meeting the gun's requirements, and notably, the bullet velocity reached 488 m/s (an 8.9% increase compared to the velocity of the currently used ammunition).

4 Conclusions

- A high-energy spherical propellant based on NM–NB was successfully developed by selecting an NM–NB/EAc ratio of 1/2.5 during the dispersion process and removing 50% of the initial solvent under controlled technological conditions. The optimized formulation yielded approximately 80% of granules within the desired particle size range of 0.125-0.315 mm, ensuring uniform combustion characteristics.
- The propellant exhibited a combustion heat of *ca.* 998 cal/g, representing a 9.07% increase compared to conventional spherical propellants. This improvement in energy content is directly associated with enhanced internal ballistic efficiency. Notably, key physical and chemical parameters, including specific gravity, bulk density, and chemical stability, remained within acceptable ranges, demonstrating that the improved formulation does not compromise safety or handling properties.
- Furthermore, coating the granules with 2% centralite and flattening them to achieve a burning thickness of 0.1-0.12 mm resulted in controlled combustion, stable chamber pressure, and improved ballistic performance. When tested with 7.62×25 mm Tokarev ammunition, a significant increase of 8.9% in muzzle velocity was observed, confirming the formulation's practical effectiveness.
- In summary, the proposed NM–NB-based spherical propellant demonstrates superior energetic performance while maintaining stability and manufacturability. The comprehensive evaluation confirms its potential as a promising candidate for small arms ammunition applications.

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Authorship contribution statement

Doan Minh Khai: conception, foundations, methods, performing the experimental part, performing the statistical analysis, other contribution to the publication
Nguyen Duy Tuan: conception, other contribution to the publication
Nguyen Tuan Anh: methods, other contribution to the publication

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