Study on the influence of compositional and technological factors on the physicomechanical and combustion properties of Nitrocellulose–Cellulose–Trinitrotoluene materials

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ABSTRACT

This paper presents the results of evaluating the influence of several factors, such as composition and manufacturing technology, on the physicochemical and combustion properties of Nitrocellulose–Cellulose–Trinitrotoluene (NC-C-TNT) materials. The findings demonstrate a pronounced effect of trinitrotoluene (TNT) content on the properties of Nitrocellulose–Cellulose (NC–C) sheets produced by the papermaking method. Increasing the amount of TNT impregnated into the NC-C sheets significantly improves the mechanical properties of the system, while also enhancing parameters such as heat of combustion, propellant force, and burning rate coefficient. However, when TNT is impregnated into NC–C sheets formed by bonding two layers of material using nitrocellulose adhesive in acetone, the aforementioned effects are not observed. The influence of technological parameters such as pressing pressure, pressing time, and pressing temperature on the properties of the material was also investigated. The results showed that these processing parameters exert a significant impact on the mechanical characteristics of the material.

Keywords: Nitrocellulose; Nitrocellulose-Cellulose-Trinitrotoluene; Compositional and technological factors; Physico-mechanical and combustion properties.

1. INTRODUCTION

At present, one of the important research directions in military science is the replacement of metallic cartridge cases and shell casings with energy-carrying composite materials [1, 2]. These materials typically comprise nitrocellulose (NC), cellulose (kraft pulp), processing additives, chemical stabilizers, and other energetic additives [3,4]. Among the energetic additives, trinitrotoluene (TNT), owing to its melting point being well-suited to the processing temperature (approximately 80 °C), is employed to impregnate Nitrocellulose–Cellulose (NC-C) base sheets, thereby producing Nitrocellulose–Cellulose–Trinitrotoluene sheets with superior mechanical properties and high energy content [4].

Internationally, Nitrocellulose–Cellulose–Trinitrotoluene (NC-C-TNT) materials have been applied in military applications; however, due to confidentiality requirements, no specific publications are available regarding the influence of compositional and technological factors on their physicochemical and combustion properties. Domestically, research and production of these materials are still in the initial stages. To enable their practical application, it is necessary to investigate and establish the effects of the aforementioned factors on the properties of the material. The objective of this study is to evaluate the influence of compositional and technological factors on the properties of NC–C–TNT sheets, thereby identifying the optimal composition and processing conditions for practical application.

2. RESEARCH METHODOLOGY

2.1. Materials and chemicals

Unbleached softwood kraft pulp imported from the United States, Cellulose content ≥ 95%;

fibrous nitrocellulose grade Pi-BA-2 with a nitrogen content of 13.21% (Z1 Factory, Vietnam); powdered trinitrotoluene (Z2 Factory, Vietnam) 99.0%; cationic starch 98.5% (MinhYang Biochemistry, Vietnam); ethanol 99.5% (Duc Giang Chemicals, Vietnam); ethyl ether 99.5% (Duc Giang Chemicals, Vietnam); and acetone 99.0% (Xilong, China).

2.2. Equipment and apparatus

PTI laboratory pulp disintegrator; PTI laboratory pulp refiner; Rapid-Köthen laboratory sheet former; 2 L three-neck round-bottom glass flask; glass beakers (250 mL, 500 mL, 1000 mL, 2 L); stainless steel trays; plastic ladles; micrometer; Binder drying oven, model ED 115, maximum temperature 300 °C; SH Scientific vacuum drying oven with adjustable maximum temperature; Ohaus PA214 analytical balance (precision 10^{-4} g, capacity 210 g); Ohaus PR2202/E precision balance (precision 10^{-2} g, capacity 2.2 kg); magnetic stirrer with contact heating up to 100 °C.

2.3. Research methodology

2.3.1. Fabrication of Nitrocellulose–Cellulose (NC–C) sheets

a. Raw material preparation

Kraft pulp in sheet form was torn into small pieces and soaked in water for approximately 24 hours. It was then disintegrated at 30,000 rpm for about 10 minutes in 5 L laboratory disintegrator, followed by refining in a PTI laboratory mill at 1,700 rpm for about 3-5 minutes. NC was air-dried for 48 hours until reaching a moisture content of about 10%, after which approximately 10 g of the sample was taken for moisture determination.

b. Component mixing

The refined kraft pulp was mixed with NC at the ratio 50:50 by mass together with 0.5% cationic starch (calculated on the total mass of NC and kraft pulp) using an IKA stirrer for no less than 15 minutes. The suspension concentration was approximately 10%.

c. Sheet formation

A Rapid-Köthen sheet former was used to produce laboratory sheets with a suspension concentration of NC and pulp in the range of 0.2% to 0.5% and a basis weight of 90 g/m². After sheet formation, the samples were dried in a vacuum dryer and then conditioned in an air-conditioned room. NC-C material samples were fabricated with NC:C-50:50 ratio, called 50:50.

d. Lamination

Dried NC with a moisture content below 0.5% was dissolved in acetone at a concentration of 1%. A thin adhesive layer (approximately 0.5 g) was applied to two dried sheets with a basis weight of 90 g/m², which were then laminated to achieve a total basis weight of 180 g/m². A light roller press was used to ensure proper bonding, after which the laminated sheets were vacuum-dried. The laminated sample is manufactured with a NC:C ratio of 50:50, called 50:50 laminated.

2.3.2. Preparation of Nitrocellulose–Cellulose–Trinitrotoluene samples

The NC–C material sheet has an approximate mass of 5.6 g was weighed to an accuracy of ± 0.02 g. A mist sprayer was used to apply a 5% stabilizer solution in ethanol onto the NC-C sheet, ensuring that the deposited stabilizer amount matched the designed value. The material was then dried at 70 °C for 2 hours. The required masses of trinitrotoluene (TNT) 5.6 g, 8.4 g, 11.2 g, and 14.0 g were weighed to an accuracy of ± 0.02 g, corresponding respectively to NC:C:TNT ratios of 50:50:100, 50:50:150, 50:50:200, and 50:50:250, then placed into a heat-resistant beaker positioned in a hot-water bath. The mixture was heated to (90-95) °C until the TNT was completely melted. The NC-C sheet was then impregnated with the molten TNT in (2-3) minutes, after which it was placed in a desiccator in an air-conditioned room.

2.3.3. Preparation of Nitrocellulose–Cellulose–Trinitrotoluene samples

The study on the influence of technological parameters on the properties of NC:C:TNT materials was conducted using a hydraulic press equipped with a mold heated by a glycerin–water solution. NC:C material samples with a 50:50 ratio and a basis weight of 180 g/m² were impregnated with 150% TNT. The mold was heated to 95 °C, and three material sheets were placed between the mold surfaces, held at the temperature for 15 minutes, and then pressed at various pressures for 15 seconds. The mechanical properties were subsequently measured.

2.3.4. Methods for technical characterization of the material

The physical mechanical strength was measured using an M350-10CT tensile and compression testing machine at a crosshead speed of 20 mm/min. The reported value represents the average of five measurements. The heat of combustion was determined in accordance with TCVN/QS 889:2019. The material's microstructure was examined using a JMS-6510LV scanning electron microscope (SEM) manufactured by JEOL, Japan.

The propellant force, covolume and burning rate coefficient we determined in a closed vessel test on B180 bomb made by HPI. The combustion parameters of the material are determined by recording the pressure–time curve in a chamber of known volume. A precisely weighed sample is ignited under controlled initial conditions. The propellant force, covolume and maximum pressure are related to each other by the formula:

$$p_m = \frac{F \cdot \Delta}{1 - \alpha \cdot \Delta} = \frac{F\Delta}{W_0 - \alpha \cdot \omega} \tag{1}$$

in which: p_m – Maximum pressure; F – Propellant force; ω – Sample mass; Δ – Packing density; W_0 – Bomb volume; α – Covolume of combustion products. To find unknown quantities, at least two tests are required. After performing them at different packing densities, two corresponding values are obtained, p_{m1} and p_{m2}. These pressure values are corrected with a coefficient for adjusting the heat transfer to the bomb wall:

$$\delta p_m = \frac{C_M}{7,774} \cdot \frac{S_b}{W_0} \cdot \frac{p_m}{\Delta} \cdot \frac{1}{100} \tag{2}$$

where C_M is the Murahur correction, %, depending on the burning time t_k; S_b - Bomb chamber surface and bomb grooves, cm^2; W_0 – Bomb volume, cm^3; 7.774, cm^2/g – Coefficient characterizing the Murahur experimental parameters. Then the covolume and propellant force are calculated according to the formulas (3):

$$\alpha = \frac{\frac{p_{m2}}{\Delta_{m2}} - \frac{p_{m1}}{\Delta_{m1}}}{p_{m2} - p_{m1}} \text{ and } F = \frac{p_{m1}}{\Delta_1} - \alpha \cdot p_{m1} = \frac{p_{m2}}{\Delta_2} - \alpha \cdot p_{m2} \tag{3}$$

The total impulse I_k, the burning rate coefficient u_1 and the half burning thickness e_1 are related to each other by the formula (4). The total impulse Ik is determined by pressure-time data, e1 is half the thickness of the material sheets.

$$I_k = \int_0^{t_k} p dt = \frac{e_1}{e_2} \tag{4}$$

3. RESULTS AND DISCUSSION

3.1. Effect of the NC:C:TNT ratio on the combustion properties of the material

The heat of combustion of combustible cartridge case (CCC) material samples with various NC:C:TNT ratios was investigated. From table 1, it can be observed that increasing the TNT content leads to a higher heat of combustion. TNT contains energy-carrying functional groups; therefore, during combustion, the additional heat released from its decomposition supplements that generated by nitrocellulose decomposition, thereby increasing the system's total heat of combustion.

Table 1. *Combustion properties of NC–C–TNT material samples with different ratios TNT, ratio of NC:C-50:50.*

Num.		**Unit**	**Ratio TNT**			
			100%	**150%**	**200%**	**250%**
1	Heat of combustion	kcal/kg	535.7	576.36	625.13	649.26
2	Volume of gas	L/kg	799.4	836.4	847.1	859.0
3	Propellant force	kJ/kg	466.059	547.542	600.125	636.275
4	Covolume	L/kg	1.42	1.33	1.30	1.28

The volume of gas generated during combustion increases with higher TNT content. TNT acts as an energetic component that drives the combustion equilibrium toward the formation of a greater amount of gaseous products, thereby resulting in a higher gas volume.

The strongest effect is observed in propellant force, with a 36.5% increase across the TNT range. Interestingly, the increments decline (81.5 kJ/kg → 52.6 kJ/kg → 36.2 kJ/kg), meaning that at higher TNT levels, the marginal ballistic benefit decreases. Nonetheless, the absolute gain remains substantial, indicating TNT is highly effective in translating chemical energy into mechanical work, especially when combined with the NC–C matrix.

Covolume decreases consistently from 1.42 to 1.28 L/kg, meaning the effective specific volume of combustion products is reduced. Lower covolume corresponds to denser gases under combustion conditions, allowing higher pressures in confined chambers. This reduction is a critical factor amplifying propellant force, even when gas volume increases are moderate.

TNT enrichment in NC–C–TNT composites leads to a systematic improvement in energy-related metrics, especially propellant force. Heat of combustion and covolume behave in complementary ways: the former increases energy availability, while the latter enhances gas compressibility, both reinforcing higher ballistic performance. Gas volume changes are moderate, indicating that TNT’s main contribution lies in energy density rather than gas yield. The most effective region of TNT addition appears between 150%–200%, where significant gains are still realized before marginal benefits decline.

For practical application, increasing TNT content beyond 200% should be carefully evaluated against safety and structural considerations of propellant chambers.

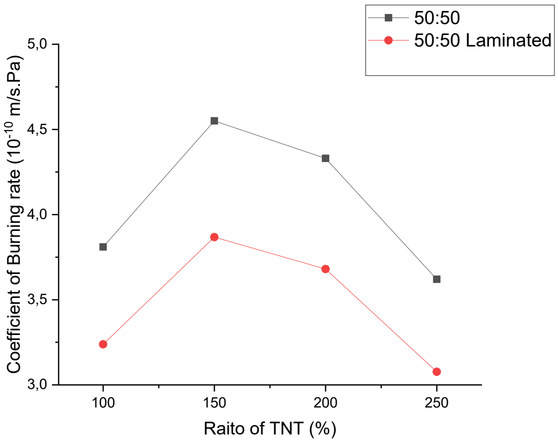


Figure 1. *Burning rate coefficient results.*

The burning rate results are presented in figure 1. When the TNT content increased from 100% to 150–200%, the burning rate coefficient rose correspondingly. However, when the TNT content exceeded 200%, the burning rate coefficient showed a slight decline. This indicates the existence of an optimal TNT content—approximately 150–200%—that maximizes the burning rate. Laminated materials demonstrated the lowest burning rate coefficient, indicating that the layered structure affects the burning rate.

3.2. Effect of the NC-C-TNT ratio on the mechanical properties of the material

Two NC-C material samples with a 50:50 ratio and a basis weight of 180 g/m² were selected; one sample was fabricated via lamination. Both were impregnated with TNT at levels of 100, 150, 200, and 250% by mass, and their mechanical properties were determined. From figure 2, it can be seen that, for the non-laminated specimens, increasing TNT content leads to increases in Young's modulus, tensile strength at break, and elongation at break. This trend can be explained by TNT filling the voids between cellulose and NC fibers; upon solidification, TNT enhances interfacial bonding within the material's structure, as is clearly evidenced in the SEM images.

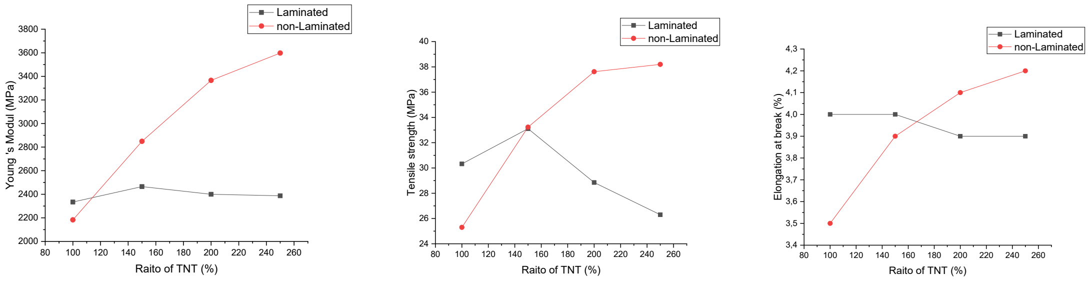
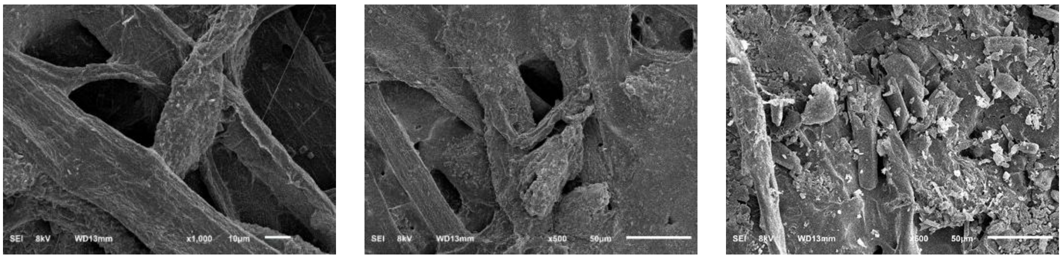


Figure 2. *Effect of the NC-C-TNT ratio on the Young's modulus, tensile strength, elongation at break of the material.*

When TNT is impregnated into a porous NC–cellulose sheet, it can seep into the spaces (voids) between the cellulose and nitrocellulose fibers. Once the molten TNT cools and solidifies, it acts like a filler and binder, strengthening the inter-fiber connections. This results in improved mechanical properties such as stiffness (Young's modulus), tensile strength, and elongation, because the structure is denser and better bonded. However, when the NC–cellulose material is first laminated using nitrocellulose adhesive, the surface becomes smoother and the fiber network is partially sealed. The lamination reduces material permeability so molten TNT cannot penetrate deeply and instead remains near the surface or in outer pores. Lack of full infiltration prevents TNT from reinforcing internal fiber bonds and can even disturb fiber–fiber adhesion, producing equal or slightly lower mechanical strength than unlaminated sheets.



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

Figure 5. *SEM image of NC-C material fabricated by the non-laminated method and impregnated with TNT at an NC:C:TNT ratio of 50:50:100 (a), 50:50:100 (b), by the laminated method and impregnated with TNT at an NC:C:TNT ratio of 50:50:150 (c).*

3.3. Effect of selected technological parameters on the properties of NC:C:TNT materials

Effect of pressing pressure on the mechanical properties of the material

The results showed that increasing the pressing pressure from 10 bar to 20 bar led to a clear increase in both Young's modulus and tensile strength. This can be attributed to the closer packing of NC and cellulose particles, resulting in reduced material thickness. However, when the pressing pressure was increased from 20 bar to 30 or 40 bar, the effect was less pronounced, likely due to the elastic deformation characteristics of the material.

Table 2. *Mechanical property test results of material samples at different pressing pressures.*

Num.	**Pressing pressure (bar)**	**Mechanical property**		
		E (N/mm^2)	**σ (N/mm^2)**	**δ (%)**
1	10	3010	35,27	4,1
2	20	3292	38,15	4,1
3	30	3341	39,01	4,0
4	40	3401	39,7	4,0

Study on the effect of pressing time on the mechanical properties of the material

The NC:C material samples were prepared at a 50:50 ratio with a basis weight of 180 g/m², and subsequently impregnated with TNT at a rate of 150%. The pressing mold was heated to 95 °C, and three sheets of the material were placed between the two mold surfaces and preheated for 15 minutes. Pressing was carried out at a pressure of 20 bar for different pressing durations. Upon completion, the samples were removed and their mechanical properties were measured to evaluate the influence of pressing time.

Table 3. *Mechanical property test results of material samples at different pressing times.*

Num.	**Pressing time (bar)**	**Mechanical Property**		
		E (N/mm^2)	**σ (N/mm^2)**	**δ (%)**
1	10	3110	34,17	4,1
2	15	3292	38,15	4,1
3	20	3315	39,51	4,0
4	25	3304	38,49	4,0

The results indicated that extending pressing time from 10 to 15 s markedly improved Young's modulus and tensile strength by densifying NC and cellulose particles, but further increases (20 - 25 s) gave little additional benefit due to elastic deformation. NC:C sheets (50:50, 180 g/m^2) impregnated with 150% TNT were pressed at 20 bar for 15 s under mold temperatures of 75 - 105 ^{o}C. Mechanical properties were poor below TNT's melting point (75 °C) and showed little improvement above it. Thus, an optimal and safe pressing range of 86-95 °C was identified, compatible with water-based heating systems.

Table 4. *Mechanical property test results of material samples at different pressing temperatures.*

Num.	**Pressing temp. (^{o}C)**	**Mechanical Property**		
		E (N/mm^2)	**σ (N/mm^2)**	**δ (%)**
1	75	2010	24,55	3,3
2	85	3270	37,57	4,1
3	95	3292	38,15	4,1
4	105	3305	38,51	4,0

4. CONCLUSIONS

This work investigates NC–C–TNT composites, focusing on how formulation and processing affect structure, mechanics, and ballistic behavior. Increasing TNT content fills microvoids in the NC-cellulose matrix, enhancing bonding and raising the heat of combustion and propellant force, while gas volume rises moderately and covolume decreases from 1.42 to 1.28 L/kg. The most effective range is 150–200% TNT, where propellant force increases by 36.5% and the burning-rate coefficient peaks; above 200% the marginal gains diminish. Laminated samples show poorer TNT penetration and the lowest burning-rate coefficients, highlighting the impact of microstructure. Processing conditions strongly influence properties: increasing pressing pressure from 10 to 20 bar and pressing time from 10 to 15 s significantly improves Young's modulus and tensile strength, but higher values provide little further benefit. Temperature is critical - below TNT's melting point (75 °C), performance is poor, while 86–95 °C ensures optimal bonding, mechanical strength, and safe operation with water-based heating systems.

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TÓM TẮT

Nghiên cứu ảnh hưởng của các yếu tố thành phần và công nghệ đến tính chất lý – cơ và tính cháy của vật liệu Nitrocellulose-Cellulose-Trinitrotoluene

Bài báo trình bày kết quả đánh giá ảnh hưởng của một số yếu tố, như thành phần và công nghệ chế tạo, lên tính chất cơ học và cháy của vật liệu Nitrocellulose–Cellulose–Trinitrotoluene (NC–C–TNT). Kết quả cho thấy hàm lượng trinitrotoluene (TNT) ảnh hưởng rõ rệt đến đặc tính của các tấm NC–C chế tạo bằng phương pháp xeo giấy. Việc tăng lượng TNT thấm vào các tấm NC–C làm cải thiện đáng kể cơ tính của hệ, đồng thời nâng cao các tham số như nhiệt lượng cháy, lực thuốc phóng và hệ số tốc độ cháy. Tuy nhiên, khi TNT được thấm vào các tấm NC-C được tạo bởi việc ghép hai lớp vật liệu bằng keo nitrocellulose hòa trong acetone, các ảnh hưởng nêu trên không xuất hiện. Các thông số công nghệ như áp suất ép, thời gian ép và nhiệt độ ép cũng được khảo sát. Kết quả cho thấy những thông số này tác động đáng kể đến các đặc trưng cơ học của vật liệu.

Từ khoá: Nitrocellulose; Nitrocellulose–Cellulose–Trinitrotoluene; Các yếu tố thành phần và công nghệ; Tính chất cơ lý và tính chất cháy.