PULSE WAVE SHAPES CHARACTERISATION IN NONTHERMAL PULSE ELECTRIC FIELD METHOD FOR FOOD PRESERVATION

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A thesis submitted in fulfilment of the requirements for the award of the degree of Doctor of Philosophy

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> > FEBRUARY 2023

DEDICATION

This thesis is dedicated to all humanity as the knowledge acquired here is beneficial to be implemented and applied for the sake of producing fresh and healthy food. It is my gift to all of us and a bet for me to face the good end of the hereafter. May Allah bless me and forgive all my sins.

ACKNOWLEDGEMENT

Many thanks to those who helped me a lot until the completion of this thesis, especially my supervisor, *Prof. Madya Dr. Mohamed Afendi*, who always gave me guidance and support in every aspect. Not to forget, my Co-supervisor, *Prof. Madya Dr. Madihah*, knows how to assist me in understanding the field in which I am no expert.

To my little family: *Muhammad Al Fatih*, *Muhammad Al Amin* and *Siti Maryam Al-Azra*' as my kids, and *Nur Ardawati* as my lovely wife, who is very understanding of the life of a Ph.D. student, I owe you my life and thanks for all the patience and support that you have poured on me. May Allah grant you a paradise in the hereafter.

Thanks to both of my parents for giving me a chance to live in this world by giving birth to me. This little effort is no comparison to the time, energy, and money that both of you have invested in raising me as a child. Without both of you, maybe I am not what I am now.

Last but not least, thanks to everyone who support me from the start, regardless of your presence in my life today. Your services will always be remembered. Nothing but a pray from me to all of you. This thesis is a sign of deep appreciation for your time with me.

ABSTRACT

Pulsed Electric Field (PEF) is a technique that does not use any heating element in inactivating food borne pathogens and spoilage microorganisms. The advantage of this technique is that it can preserve the physical properties of food, such as colour, flavour, and nutritional value while extending its shelf life. With the straightforward working principle and well-known advantages, this technique can replace the traditional method for food pasteurisation which uses heat. However, existing PEF devices lack several features: low voltage amplitude, limited control of pulse properties, and expensive besides no optimal range of treatment parameters clearly defined for specific foods. These shortcomings limit their function in the subset of pulse applications. Therefore, this study was conducted to fill the available innovation space to solve or at least reduce the shortcomings in PEF devices today. In this thesis, a compact high-voltage pulse generator was developed by implementing the concept of capacitor discharge to produce a square pulse (mono polar fashion) to inactivate microbes inherently in raw goat's milk. The development phase began with a simulation study regarding the design of a compact high voltage pulse generator circuit and a treatment chamber that produced the desired results. Then, it was tested practically on sodium chloride (NaCl) solution with the conductivity of 100 mS/m, and the result was the same as the simulation with few insignificant differences. From the experiments performed on raw goat's milk, the results obtained suggest that a frequency of 10 Hz, a voltage amplitude of 4 kV, and a pulse width between $1 - 4 \mu s$ promise the best results in the treatment process. The inherent microorganisms have been successfully reduced from 2.98×10^6 Colony Forming Unit (CFU)/ml to 1.64×10^6 CFU/ml, an almost 60% reduction or 0.55 survival ratio. This thesis also suggests that the compact high-voltage pulse generator has a vast potential to inactivate other types of microorganisms in various kinds of liquid food. This is due to the flexibility and reliability offered by the compact high voltage pulse generator developed in this study.

ABSTRAK

Medan Elektrik Terdenyut (PEF) merupakan teknik yang tidak menggunakan sebarang unsur pemanasan dalam menyahaktifkan patogen bawaan makanan dan mikroorganisma pembusuk. Kelebihan teknik ini ialah ia dapat mengekalkan sifat fizikal makanan, seperti warna, rasa, dan nilai pemakanan disamping memanjangkan jangka hayatnya. Dengan prinsip kerja yang mudah dan kelebihan yang telah dikenali, teknik ini boleh menggantikan kaedah tradisional untuk pempasteuran makanan yang menggunakan haba. Walau bagaimanapun, peranti PEF sedia ada kekurangan beberapa ciri: amplitud voltan rendah, kawalan sifat dedenyut yang terhad, dan mahal selain tiada julat optimum parameter rawatan yang ditakrifkan dengan jelas untuk makanan tertentu. Kelemahan ini mengehadkan fungsinya dalam subset aplikasi dedenyut. Oleh itu, kajian ini dijalankan bagi mengisi ruang inovasi yang ada untuk menyelesaikan atau sekurang-kurangnya mengurangkan kekurangan pada peranti PEF yang sedia ada. Dalam tesis ini, penjana dedenyut voltan tinggi padat telah dibangunkan dengan melaksanakan konsep nyahcas kapasitor untuk menghasilkan dedenyut persegi (fesyen monopolar) untuk menyahaktifkan mikrob yang wujud dalam susu kambing mentah. Fasa pembangunan dimulakan dengan kajian simulasi berkaitan reka bentuk litar penjana dedenyut voltan tinggi padat dan ruang rawatan yang menghasilkan keputusan yang diinginkan. Kemudian, ia diuji secara praktikal pada larutan natrium klorida (NaCl) dengan kekonduksian 100 mS/m, dan hasilnya adalah sama dengan simulasi, cuma dengan sedikit perbezaan yang tidak ketara. Daripada eksperimen yang dilakukan terhadap susu kambing mentah, keputusan yang diperoleh menunjukkan bahawa frekuensi 10 Hz, amplitud voltan 4 kV, dan lebar dedenyut antara 1 – 4 µs menjanjikan hasil terbaik dalam proses rawatan. Mikroorganisma yang wujud telah berjaya dikurangkan daripada 2.98×10^6 Colony Forming Unit (CFU)/ml kepada 1.64×10^6 CFU/ml, pengurangan hampir 60%. Tesis ini juga mencadangkan bahawa penjana dedenyut voltan tinggi padat ini mempunyai potensi besar untuk menyahaktifkan jenis mikroorganisma lain dalam pelbagai jenis makanan cecair. Ini disebabkan oleh fleksibiliti dan kebolehpercayaan yang ditawarkan oleh penjana dedenyut voltan tinggi padat yang dibangunkan dalam kajian ini.

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LIST OF ABBREVIATIONS

CAD	-	Computer-Aided Design		
CFU	-	Colony Forming Unit		
CMOS	-	Complementary Metal Oxide Semiconductor		
FEA	-	Finite Element Analysis		
FEM	-	Finite Element Method		
HPP	-	High-Pressure Processing		
IGBT	-	Insulated Gate Bipolar Transistor		
MOSFET	-	Metal-oxide Silicon Field Effect Transistor		
OCnA/B	-	Output Compare A/B		
OCRnA/B	-	Output Compare Register A/B		
PARDISO	-	Parallel Direct Solver		
PCB	-	Printed Circuit Board		
PDE	-	Partial Differential Equation		
PEF	-	Pulsed Electric Field		
PFN	-	Pulse Forming Network		
PWM	-	Pulse Width Modulation		
Re	-	Reynolds Number		
TCCRnA/B	-	Timer/Counter Control Register		
TCNTn	-	Timer/Counter		
TFTC	-	Too Few To Count		
TNTC	-	Too Numerous To Count		
TTL	-	Transistor Transistor Logic		

LIST OF SYMBOLS

$\Delta \varphi$	-	Transmembrane Potential
μ	-	Dynamic Viscosity
A	-	Effective Area of The Electrode
С	-	Capacitance
C_{DG}	-	G-D Capacitance
C_{GS}	-	G-S Capacitance
C _{JO}	-	Junction Oxide Capacitance
C_m	-	Capacitance of The Membrane per Unit Area
C_P	-	Specific Fluid Heat Capacity
d	-	Diameter
Ε	-	Electric Field Strength
F	-	Microorganism Shape Factor
f	-	Pulse Frequency
g	-	Gravitation Accelaration Constant
Ι	-	Current Flows
I_D	-	Drain Current (Continuous)
I_{DP}	-	Drain Current (Pulse)
I_S	-	Leakage Current
k	-	Thermal Conductivity
K_P	-	Transconductance
k_T	-	Turbulent Thermal Conductivity
l	-	Length of The Treatment Area
lambda	-	Channel Length Modulation
М	-	Junction Grading Coefficient
n	-	Number of Pulses
Ν	-	Number of Colonies After PEF Treatment
N_o	-	Number of Colonies Before PEF Treatment
Р	-	Pulse Power
P_e	-	Electrocompressive Force
P_m	-	Mechanical Restoring Force
Q	-	Charge Accumulated
Q_{pulse}	-	Each Pulse Energy Delivered
Q_{total}	-	Total Pulse Energy Delivered

r _c	-	Cell's Radius
R_{ch}	-	Treatment Chamber Resistance
R_{DS}	-	On Resistance
R_s	-	Switch Resistance
R_t	-	Total Resistance
S_x	-	Fraction of Surviving Cells
t	-	Time
v	-	Fluid Flow Velocity
V	-	Voltage Applied
V_{ch}	-	Voltage Across Treatment Chamber
V_{DS}	-	Drain – Source Voltage
V_J	-	Junction Voltage Drop
V_{TO}	-	Zero Bias Gate Threshold
W	-	Energy Stored
Y	-	Elastic Modulus of The Membrane
δ	-	Thickness of The Membrane Cell
δ_0	-	Initial Membrane Thickness
${\cal E}_0$	-	Permittivity of Free Space
\mathcal{E}_m	-	Relative Permittivity of The Cell Membrane
E _r	-	Relative Permittivity of The Dielectric Material
θ	-	Angle
ρ	-	Fluid Density
σ_e	-	Electrical Conductivity of The External Medium
σ_i	-	Electrical Conductivity of The Cytoplasm
σ_m	-	Electrical Conductivity of The Cell Membrane
τ	-	Pulse Width
$ au_m$	-	Characteristic Time Constant
arphi	-	Multiplication Factor

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CHAPTER 1

INTRODUCTION

1.1 Background Study

In food processing, pasteurisation kills pathogens and extends shelf life by using moderate heat, usually less than 100 °C. Through this process, all organisms and enzymes liable to contribute to food spoilage or foodborne illness, including vegetative bacteria, are killed (or destroyed if possible) (Tewari and Juneja, 2008; Fellows, 2009). It is undeniable that there are advantages to the process. However, there are also disadvantages, as processed food changes its physical properties, such as colour, flavour, and nutritional value (Lado and Yousef, 2002; Toepfl *et al.*, 2006; Kishore *et al.*, 2007; Chauhan and Unni, 2015).

In addition to the problems faced, consumer awareness of food content and processing methods also contributed to searching for new food treatment approaches (Evans and Cox, 2006; Mohamed and Eissa, 2012). As a result, electrical engineers and food technologists collaborate to develop alternative methods that remove thermal elements involved in food processing to resolve the stated problems and ensure that consumers get healthier and fresher food. Therefore, a nonthermal technique approach is proposed.

Several nonthermal techniques have been studied recently because of their potential for inactivating pathogenic microorganisms. It is therefore explored globally, causing research on it to increase. Ultimately, all of this is done to ensure consumers are provided with a microbiologically safe product. Table 1.1 shows some of the known nonthermal methods with their comparisons that have prospective as an alternative to conventional methods.

Tashnalagu	Intensity -	Type of food		Annuoval	Defense
Technology		Solid	Liquid	Approval	Reference
Pulsed electric field	5-55 kV/cm	Beef muscles, potato tubers	Fruit juice, liquid eggs, milk	Limited approval in the US (FDA, no objection letter from 07/07/1995)	(Faridnia, 2015; Qin <i>et al.</i> , 1996)
High-pressure processing	100-1000 MPa	Ham, seafood	Fruit juice, guacamole, jam, salad dressing, milk	Japan, North America, Europe	(Hoover <i>et al.</i> , 1989; Smelt, 1998)
Ultraviolet radiation	0.5-20 J/m ²	Meat surface, shell egg surface	Orange juice	Approval pending in the US	(Bintsis <i>et al.</i> , 2000; Kuo <i>et al.</i> , 1997)
Gamma irradiation	2-10 kGy	Raw poultry meat, raw	Liquid eggs	More than 41 countries	(Farkas, 1998; Agrawal and Goyal, 2017)

 Table 1.1
 Summary of known nonthermal methods by comparison

The nonthermal methods showcased in Table 1.1 are recognised as a novel process due to their ability to inactivate microorganisms at ambient, sub-ambient, or slightly above ambient temperature (Butz and Tauscher, 2002; Kumar *et al.*, 2016). These techniques are specifically designed to eliminate thermal components in processing while maintaining food's flavour, appearance, and nutritional value (Barbosa-Canovas *et al.*, 1999; Jambari, 2014).

Among the nonthermal methods presented here, the pulsed electric field (PEF) appears to be the most extensively studied in addition to high hydrostatic pressure (HPP) and high-intensity ultrasound combined with pressure. It is due to the short treatment time, which reduces the heating effect compared to other technologies. On the other hand, regarding gamma irradiation, although it has a high potential for commercialisation, this method has been hampered by the bad perception of society in the past (Resurreccion *et al.*, 1995).

1.2 Overview of Pulsed Electric Field

The PEF approach has been used in studies since the 1960s to take advantage of its influence on the inactivation of microorganisms, mainly bacteria found in liquid foods. On the other hand, Sale and Hamilton were among the first to give a thorough and scientific study of this method in a series of papers published in 1967 and 1968 (Sale and Hamilton, 1967; Hamilton and Sale, 1967; Sale and Hamilton, 1968). Later, Zimmerman and his colleagues expanded on this work by establishing the theory of dielectric rupture and publishing several papers (Coster and Zimmermann, 1975; Zimmermann *et al.*, 1976). Their research was performed in the 1970s at the University of Wurzburg in Germany.

Since then, scholars have become increasingly passionate about PEF research, gaining traction. This topic rapidly grew in popularity, with published papers delivering discussion after discussion. The study of PEF treatment technology encompasses a wide range of subjects, including the characteristics of suitable microorganisms, the construction of modern electroporators and their deployment in the industry, as well as food safety policies (Campbell *et al.*, 2008; Reberšek and Miklavčič, 2011b; Toepfl, 2011; Stankevič *et al.*, 2013; Raso *et al.*, 2016).

The fundamental principles of PEF technology can be defined by applying short electrical pulses to food located between electrodes separated by an insulator. The electrical pulses can range between microseconds to milliseconds and typically yield an electric field intensity of 10 - 80 kV/cm. This approach is commonly used to treat liquid foods, but there is no doubting that it may also be used to treat semi-solid and solid foods (Ramaswamy *et al.*, 2005).

PEF technology is more suitable for pasteurizing liquid foods such as milk, juices, yoghurt, soups, and liquid eggs (Vega-Mercado *et al.*, 1997; Bendicho *et al.*, 2003; Puc *et al.*, 2004). It is because of the fluid properties that can transfer electricity due to the presence of several ions and causing it to obtain a certain degree of electrical conductivity. Therefore, the electric current flowing is scattered to every point in the liquid due to the presence of the charged molecules (Zhang *et al.*, 1995).

PEF treatment involves a phenomenon known as electroporation or electropermeabilisation, which is the permeability of cell membranes as a result of being subjected to an electric field (Jordan *et al.*, 2013). If the applied field strength exceeds the critical field strength, it may experience cell disintegration; otherwise, it may revert to its original state. These circumstances could have both reversible and irreversible effects on the cell membrane, demonstrating that the impact of PEF treatment can be well controlled depending on its application by accurately altering the field strength.

The benefits of using PEF treatment technology are no longer foreign as it can preserve a high-quality fresh-like natural flavour, high nutritional value, and extend shelf life (Castro *et al.*, 1993; Qin, 1995). It is achieved by eliminating pathogens and spoilage microorganisms without using thermal elements. However, some foods, such as fruit juice, must be refrigerated after being treated to retain their aroma and flavour and increase their shelf life (Ramaswamy *et al.*, 2005; Jambari, 2014).

Studies also show that PEF treatment techniques can reduce energy consumption by 10% for each treatment compared to thermal processing (Charles-Rodríguez *et al.*, 2007). This scenario occurs because the temperature increase of each treatment is minimal and is highly dependent on the total pulse delivered, pulse width, frequency, and velocity of the food flow (in a continuous system). This 10% reduction in energy use may pique the interest of industry participants because it is a source of profit for them in the long run and may reduce manufacturing expenses. As a result, PEF-treated food can be sold at competitive rates.

1.3 Problem Statement

Implementing PEF in inactivating microorganisms is now a phenomenon and attracts researchers' interest. This method causes minimal impact on the physical properties of food; among them are flavour, colour, and nutritional value. In addition, it causes the quality and freshness of the food to be well preserved. With all its advantages, it is seen to be raised as an alternative way to replace conventional food treatment methods that use heat as the main instrument. To achieve this, it is inevitable to have a high-powered pulse generator capable of producing consistent pulses whose properties can be controlled.

Pulse generators available today consist of two types of power electronic converter circuits: conventional and non-conventional. Transmission lines and PFNs are among the popular conventional circuits while Multi Stacked MOSFETs and Cascade H-Bridge Multilevel Inverters are among the emerging non-conventional techniques for generating high voltage pulses (Baker and Johnson, 1993; Roodenburg et al., 2005; Sun and Wang, 2014; Jambari, 2014). However, most of the proposed circuits implement many components which end with complex system of circuitry and control. Some of it use more than one MOSFET to generate high voltage and high current pulses which results in intricate triggering algorithm and employing numerous MOSFET's drivers. Not only that, the use of transmission lines and spark gaps restricts the flexibility to produce a wide range of pulse widths and slow switching which constrain the generation of nanosecond pulse. The use of transformers to obtain high voltage limit the compactness of the pulse generator, thus, result in expensive, heavy, and bulky generator as well as consume more space. Furthermore, employment of transformers requires maintenance from time to time and increasing the system power loss due to higher number of winding coils.

Referring to the stated problem, this study proposes the use of capacitordischarge technique with MOSFET implementation for fast switching and microcontroller to generate flexible pulse properties. This suggested solution can improve the ability of PEF treatment to achieve effective food pasteurisation while also being able to change pulse properties to achieve desired pulse specifications for specific pulse applications. In addition, this capacitor discharge technique uses less components which makes it easier to control and does not require a complex algorithm because a single high-power MOSFET unit is implemented. As well, this method does not require transformers to obtain high voltage pulses because it can be achieved using flyback transformers that are small but high durability and do not require maintenance yet easy to control. Therefore, the finish product can be compacted, low power consumption, low cost, and flexible compared to transmission line, PFN, Multi Stacked MOSFETs, and Cascade H-Bridge Multilevel Inverters techniques. Non-complex circuit layout allows it to be comfortably packed, easy to install and can have portable features.

1.4 Research Objectives

The objectives of the research are:

- (a) To develop a compact high-voltage pulse generator using a capacitor discharge approach to generate complete control over square wave pulse properties for PEF treatment applications.
- (b) To verify the operation of the whole system of the pulse generator via simulation and hardware implementation to obtain optimum development by reducing the effect of parasitic circuit elements.
- (c) To analyse the performance of the compact high voltage pulse generator in delivering the intended pulse properties and inactivating pathogens and spoilage microorganisms present in raw goat milk including characterizing the best pulse parameters for it.

1.5 Scope of Research

This research highlights the overall development of a compact high voltage pulse generator using the capacitor discharge method. Despite underutilised components, this method also can guarantee the generation of a square pulse whose properties can be controlled accordingly - the width and frequency can vary in a broad domain selection. Square pulse in pulsed electric field treatment technology is superior due to its significant advantage in inactivating microorganisms than other pulse forms such as exponential decay and oscillation.

This study considers the implementation of microcontrollers regarding pulse signal controllers. The well-known advantages and low price are the main reasons it is chosen. With suitable programming methods and algorithms, it can generate pulse signals that can be controlled easily and systematically. In addition, the generated pulse signal is TTL and CMOS compatible, which can be used to drive any solidstate switching device such as IGBTs and MOSFETs. Since the production of nanosecond pulses requires a fast-switching device, the implementation of MOSFETs is employed.

The PEF treatment system is incomplete without the treatment chamber. A treatment chamber is a place where food is treated. It can be batch or continuous; though, continuous is more suitable for implementation in the industry due to production demands. However, the treatment chamber is not merely for food accommodation but also to house electrodes. The electrode arrangement may be in the order of parallel, coaxial, or collinear. Usually, they are positioned in parallel to obtain the most uniform electric field distribution compared to coaxial and colinear.

The treatment media or the food to be treated habitually comes in a liquid form. It does not deny that this PEF method can treat semi-solid and solid foods, but it works better with liquid food. The liquid owns a certain degree of electrical conductivity due to the presence of several ions, which result in the flow of electrical current to every point in it. Therefore, this research appointed raw goat's milk as a test subject. However, no specific microorganisms have been identified for inactivation but rather to kill any pathogens and spoilage microorganisms so that it is safe to consume.

1.6 Research Significant

PEF treatment technology has demonstrated its ability and reliability in killing the vegetative cells while colours, flavours, and nutrients are well preserved. Besides, there is no evidence of toxicity involved in using this method. Meanwhile, it consumes a relatively short treatment time to treat the media. Also, it can be used to decontaminate heat-sensitive foods suitable for the type of liquid diet. Research has shown beyond doubt about the pasteurisation of fruit juices, soups, liquid eggs, and milk. The most important thing is it offers no environmental hazard.

This study's result will help promote the implementation of PEF treatment technology on an industrial scale. The primary concern of industrial consortium interested in PEF implementation is initial investments. This issue is one of the most important topics. To the present day, only several countries adopt the PEF method in producing safe, nutritious, and high-quality products, such as Pure Pulse Technologies, USA, and Thomson-CSF, France. Therefore, this study aims to reduce its development cost while optimizing the critical process factors.

1.7 Contributions to Knowledge

This research contributes to a wide range of knowledge as it incorporates several disciplines of expertise, including high voltage and high current, power electronics, and food technology.

From the point of view of high voltage and high current fields, it contributed to the development of compact high voltage pulse generators that can be used in various pulse applications. In addition to low development costs and reliable results, it is also successfully compacted so that it can be taken anywhere. The success of developing this compact high voltage pulse generator is a superior manifestation of how high voltage and high current are well regulated.

Whereas from the point of view of the field of power electronics, it shows success in generating pulse signals whose properties can be modified as needed. The use of a microcontroller along with its complex programming algorithms has been successfully utilised to change the properties of a pulse in terms of its pulse width and frequency. Pulses as small as 63 ns and as wide as 4 ms with frequencies as low as 1 Hz and as high as 1 kHz have been successfully generated stably.

Finally, the point of view is taken from the food technology field. In this field, the studies are conducted to contribute to the production of food treatment technologies that do not use heat. Furthermore, this developed technology also provides benefits in understanding the response of microorganisms when exposed to a pulsed electric field which helps to develop further the mechanism of dielectric

breakdown theory for better understanding. The ultimate result is to produce food that is high quality, fresh, and lasts longer for the good of universal humanity.

1.8 Thesis Organisation

Chapter 2 consists of a literature review related to the application of PEF, including microbial inactivation mechanisms, and critical factors. In addition, it also touches on PEF system components, various PEF generator techniques, and commercial electroporation instruments. Thus, it covers almost essential topics associated with PEF treatment technology.

Chapter 3 defines the development process of the compact high-voltage pulse generator based on the capacitor-discharge concept. Also, it explains in detail the suitable component used to develop the electroporator and proposed circuit design to ensure the reliability and stability of the output pulse.

Chapter 4 describes the results obtained through simulations and practical studies regarding the performance of the developed compact high-voltage pulse generator. For the actual experiment, raw goat's milk from Osman Goat Farm was treated via PEF. The results obtained were analysed to observe the effectiveness of the PEF technique in inactivating the inherent microorganisms.

Chapter 5 defines the possible future work that can be done to uplift and enhance the application of PEF technology especially in industrial sector. It discusses more on the improvement of the technology itself like changing in treatment chamber design and encouraging the application of nanosecond pulse.

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- Microcontroller-based pulse signal controller development for compact high voltage pulse generator: Practical development in food treatment technology NF Kasri, MAM Piah, A Hamzah, Z Adzis Journal of Food Processing and Preservation, 45(6), p.e15531
- 3. Compact high-voltage pulse generator for pulsed electric field applications: Labscale development

NF Kasri, MAM Piah, Z Adzis

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4. Development of compact pulse generator with adjustable pulse width for pulse electric field treatment technology

NF Kasri, MAM Piah

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5. A compact and reliable pulse generator using dual 555-timer IC to produce PWM method

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